CETEM

CENTRO DE TECNOLOGIA MINERAL
MINISTÉRIO DAS MINAS E ENERGIA
(CONVÊNIO DNPM/CPRM)

TECNOLOGIA DE FORNOS DE LEITO FLUIDIZADO

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NOTA

I99 I/2004

A FIM DE POSSIBILITAR O ACESSO MAIS RÁPIDO ÀS INFORMAÇÕES CONTIDAS NESTA PUBLICAÇÃO, CUIDADOS DE REVISÃO FORMAL FORAM NEGLIGENCIADOS, SENDO DA TOTAL RESPONSABILIDADE DO(S) AUTOR(ES) A QUALIDADE DO TEXTO APRESENTADO.

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SECRETARIA DE TECONOLOGIA INDUSTRIAL - STI, do
MINISTÉRIO DE INDUSTRIA E COMERCIO - MIC
em Cooperação com a

AGÊNCIA CANADENSE DE DESENVOLVIMENTO INTERNACIONAL
(CIDA)

Realizado no período outubro/81 a março/82 no
CANADA CENTRE FOR MINERAL AND ENERGY
TECNOLOGY - CANMET na Cidade de
Ottawa, CANADÁ

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Science and Technology
Science et Technologie

CANMET
555 Booth Street
Ottawa, Ontario
K1A 0G1

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October 16, 1981

CIDA/CANMET TRAINING PROGRAM: FLUIDIZED BED TECHNOLOGY

JORGE L.L. BRANDAO - CENTRE FOR MINERAL TECHNOLOGY (CETEM), BRAZIL

Program description and chronogram:

1. Visits to select non-ferrous smelters and industrial pilot and laboratory facilities will be undertaken to observe the operation of fluidized beds, to collect data and to correlate the data among laboratory, pilot and commercial size reactors. Inco, if they will release data, could be a valuable source. Visits to equipment manufacturers, consulting firms and the New Brunswick Research and Productivity Council would also be useful. A summary report would be produced.

2. Having acquired an understanding of the important design and operating parameters and, hopefully, a correlation factor for upscaling of reactors, a continuous feed/discharge unit with the auxilliary equipment (shown in Figure 1) should be constructed. The unit must be capable of operating at 1100°C. Obviously equipment components will be obtained concurrently with "1"; the purchase of a small-scale Esp would likely be necessary.

3. Conduct roasting tests employing a Canadian zinc concentrate(s) similar to ones which would be encountered in Brazil. Various operating parameters and variations in feed composition will be tested. The results should be compared to available data on commercial operations as obtained in "1" above. Subsequent test work could be done to monitor minor element (Hg, As, Sb, etc.) behaviour during fluid bed roasting. This would be within the mandate of the project on "Control of Noxious Pyrometallurgical Emissions".

4. If a new project on silver behaviour in the zinc industry is approved, the fluid bed apparatus could also be employed to monitor silver deportment during zinc concentrate roasting. Test variables would include silver mineral form and concentration, the effect of various forms and concentrations of chloride, the presence of selenium, etc. Efforts would be made to correlate the test data to the commercial results for silver distribution in the zinc industry.

5. Simultaneously, thermodynamic calculations will be carried out to predict the equilibrium behaviour during reaction; e.g., sulphate formation, equilibrium species of minor constituents under various operating conditions, SO_2/SO_3 formation etc.

6. Efforts will be made to develop a computer model to predict the behaviour of a fluidized bed reactor that would have the capability of estimating accurately the effect of various operating and/or design parameters on the throughput, composition of the product, composition of the off-gas and overall heat and mass balances. This is an extensive program in itself, and it is assumed that only a good start will be made in this area.

The above program, if extensively carried out, will be beneficial to Mr. Brandao, to Brazil and to CANMET. For the visiting engineer, it offers a challenging program in an area of personal interest, and will allow him to return to Brazil to set up a fluid bed test facility with direct relevance to commercial fluid bed technology. The program will have immediate benefits to CANMET programs and expertise, and shall transfer many of these benefits to Brazil when Mr. Brandao returns home. Some of the specific advantages to be gained by CANMET, and later by Brazil are:

(a) Development of expertise within CANMET and the Brazilian Centre for Mineral Technology on fluid bed technology.

(b) Availability of a laboratory scale fluidized bed reactor which could be used to study scrubbing of mercury from pyrometallurgical off-gases, combustion of coal, roasting of metal sulphides, dechlorination of ferric chloride, reduction of metal oxides, etc.

(c) An understanding of the distribution of elements, including the toxic ones, during fluidized bed roasting of sulphide concentrates; also the interrelationship between the various minor elements present and the roasting parameters necessary to recover the main metals of interest.

(d) Development of a computerized model for predicting the behaviour of a fluidized bed reactor.

(e) Clarification of upscaling parameters for fluidized bed reactors.

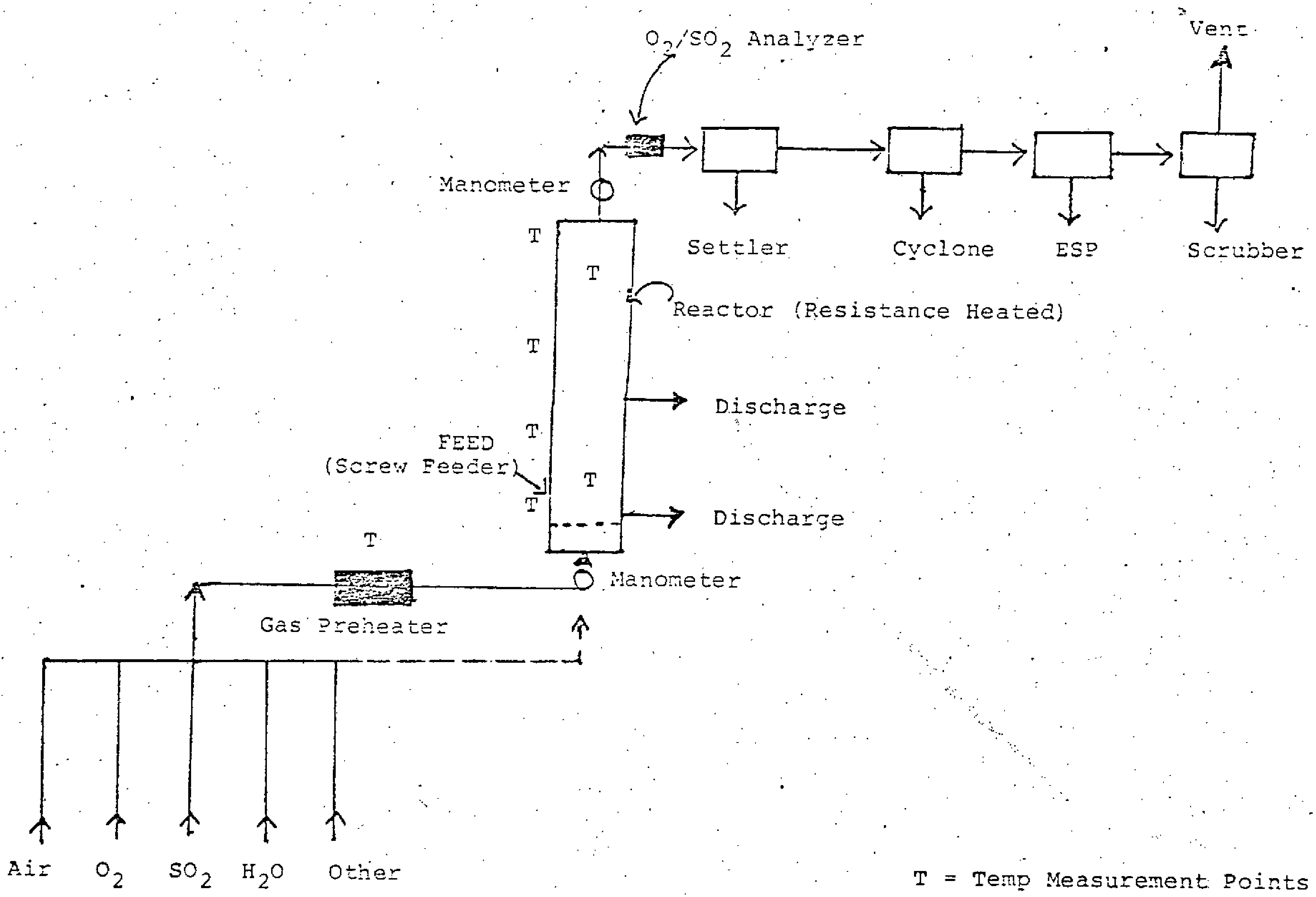
Ottawa, October 16, 1981

Dr. Peter Pint,
Program Supervisor.

Dr. J.E. Dutrizac,
J E Dutrizac
Head of the Metallurgical Chemistry Section.

PP/JED/jo

cc M. Campbell



T = Temp Measurement Points

Figure 1

- PROGRAM CHRONOGRAM -

	October				November				December				January				Febuary				March				Estimated % Completion
Visits & Data Correlation (1)				▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	80
Construction of Equipment			▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨											100
Roasting Tests & Calculation (2)														▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	60
Elaboration of Computer Model					▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	30
Progress Report												▨											▨	▨	100
Final Report (3)																							▨	▨	100

OBSERVATIONS:

(1) Visits will include a three-day course on fluid-bed reactors given by Dr. Frederick A Zenz in New Orleans, U.S.A., and other courses or symposiums of interest. Plant visits as well as visits to other research facilities, equipment manufacturers and consulting firms will be extensive throughout the program in order to collect data and to correlate them with lab. tests.

(2) Calculations include a theoretical follow-up of the process.

(3) A final report will be written at this point, unless the training program can be extended to its completion (except for the computer model which may continue after the end of Mr. Brandao's training period).

Relatorio Mensal de Andamento ao CETEM

DIVISAO: DIMETE DATA: 30 de outubro de 1981

Projeto: Estagio STI/CIDA - Tecnologia de Fornos de Leito Fluidizado
Canadian Centre for Mineral and Energy Technology - CANMET
555 Booth Street, Ottawa, ONTARIO, K1A 0G1, CANADA

Relatorio no. 01

De: Jorge L.L. Brandao,
CANMET, Room 270.

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

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Relatorio no. 1: Outubro/1981

No decorrer do mes de outubro foram realizadas as seguintes actividades:

1. Preparaçao do programa de estagio
2. Aprovaçao do programa e elaboraçao do cronograma de actividades (copias enviadas às entidades interessadas).
3. Contatos com entidades de pesquisa e industrias referente à visitas técnicas.
4. Aprovaçao, junto à CIDA, de um curso de 3 dias em tecnologia de fornos de leito fluidizado, a ser realizado de 9 a 11 de novembro em Nova Orleaés, EEUU. (Copia do programa do curso e outros detalhes em anexo).
5. Preparaçao de equipamento de estudo para testes a frio e a baixas temperaturas, em reator de "pyrex". Testes de verificaçao até 160°C com concentrado de zinco umido e seco.
6. Modificaçoes no tubo reator e testes em batelada. Colocaçao de termopar para tomar a temperatura do leito.
7. Determinaçao da taxa de vazao, pontos de amostragem, alimentaçao, vazao do produto e arraste.
8. Determinaçao da TDH (Transport Disengaging Height) para tomada do produto de arraste.
9. Acomplamento de camara de descompressao e ciclone para coletar os produtos de arraste.
10. Inicio dos testes continuos no equipamento de estudos.

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555 Booth Street,
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Programa de actividades fara o proximo mes - Novembro/1981:

1 - Continuaçao dos testes no equipamento de estudos.

2 - Curso técnico em Nova Orleaes, EEUU: 9-11/11.

3 - Visitas técnicas a centros de pesquisa e industria:

  i) Ontario Research Foundation, Toronto, Ontario,

  ii) Inco Research Labs., Sheridon Park, Mississauga, Ontario,

  iii) Falconbridge Research Labs., Thornhill, Ontario,

  iv) New Brunswick Research & Productivity Council, Frederickton, New Brunswick,

  v) Gaspé Mining & Smelting (Noranda Group), Murdochville, Quebec,

  vi) CEZ (Canadian Electrolytic Zinc, Noranda Group), Valleyfield, Quebec.

4 - Confecçao do equipamento definitivo para testes com concentrados de zinco e outros sulfetos.

5 - Tentativa de inicio do modelo para computador.

6 - Continvaçao dos testes com o equipamento de estudo, introduzindo provaveis modificaçoes trazidas pelas visitas técnicas e curso.

Ottawa, 30 de outubro de 1981

Jorge L.L. Brandao

Jorge L.L. Brandao
eng. metalurgico

JLB/jo

AIChE Today Series

Detroit, Montreal, New Orleans

FLUID BED REACTORS
ESIGN, SCALEUP, PROBLEM AREAS

Detroit, Wednesday to Friday, August 19-21, 1981

Montreal, Monday to Wednesday, October 5-7, 1981

w Orleans, Monday to Wednesday, November 9-11, 1981

An important area of chemical engineering that has gained increasing prominence in recent years is fluidization technology. It is utilized in numerous industrial applications including solids ansport, multiphase contracting, thermal and kinetic processes. o successfully design and operate fluidized systems, the acticing chemical engineer relies on a judicious balance of practical experience coupled with an understanding of the fundamentals.

This course will emphasize the basic physical phenomena nd the practical approach to design in sizing fluid bed reactors; imensioning the grid, estimating bubble size and rate of rise, calculating entrainment, choosing, locating, hanging and alculating the efficiency of cyclones and similar recovery uipment, estimating the optimum particle size and maximum ubble diameter, sizing internal heat recovery coils; estimating the size of equivalent transfer line reactors, and calculating line losses and pressure balances. Though some derivations will be eviewed they will be simple and more for background purposes to lustrate the procedure and the rationale for simplification.

The course is intended for process and design engineers, technical service groups, general troubleshooters, plant operating group leaders and most any personnel involved in andling particulate materials especially if connected with a fluid ed plant.

COURSE OBJECTIVES

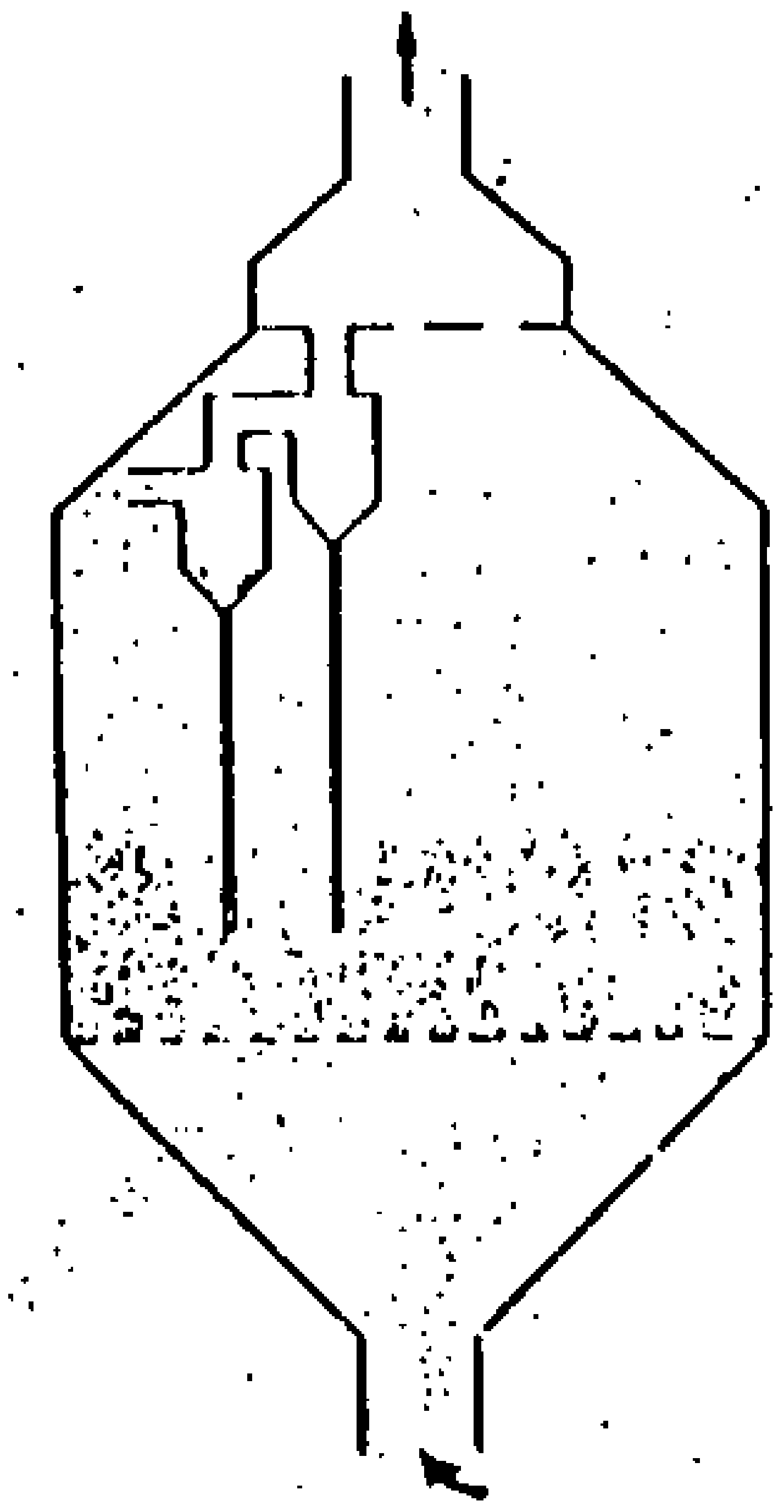
Upon completion of his course the attendees should be miliar with the background and derived relationships for calculat- ng:

(a) Gravity, pressured, and fluidized flow rates of particle beds through standpipes and orifices.
b) Incipient fluidization velocities, bed expansion, particle entrainment rates, and transport disengaging heights.
c) Initial and maximum bubble sizes, merger rates, and grid characteristics.
(d) The relative degree of gas catalyst contact to be anticipated in vessels of given dimensions.
e) The pressure drop in dense and dilute phase pneumatic conveying.
(f) The design of cyclonic particle recovery equipment.

ECTURER

The lecturer in this AIChE Today Series will be Dr. Frederick A. Zenz, a private consultant for the past 19 years, a Professor of Chemical Engineering at Manhattan College, and Technical irector of Particulate Solid Research, Inc., a non profit industry- ponsored research company engaged in relatively large scale xperimentation in fluid beds, conveying, particle attrition, particle recovery and associated fields. He is also vice-president of Engineering at the Ducon Co., Mineola, L.I., a division of U.S. ilter Corp.

Dr. Zenz received his Bachelors degree in chemistry from ueens College, the M.Ch.E. from New York University, and the Ph.D. from what is now the Polytechnic Institute of New York. rior to 1962 he spent a total of twenty years in industrial plant esign and development with the M. W. Kellogg Co., Hydrocarbon esearch Inc., and a subsidiary of Stone & Webster Engineering Corp.



COURSE OUTLINE

- **First, Second, and Third Day—9:00 A.M.-5:00 P.M.**

I. Critical Areas in Fluid Bed Design and/or Scale up
  1 Bed Solids
  2 Grid
  3 Bubbles
  4 Entrainment
  5 Recovery

II. Bed Solids
  A. Incipient fluidization
    1 Bed friction
    2 Incipient buoyancy-incipient bubbling
    3 Bed expansion and terminal velocity
    4 Correlations of incipient fluidization
    5 Empirical correlation of bed expansion
  B Bulk solids flowability
    1 Characteristic angular properties of bulk solids
    2 Efflux rates-gravity and fluidized flow
    3 Bed support experiments

III. Grid Design
  A Bubble formation at grid ports
  B. Gas distribution over grid areas
  C. Proximity of grid holes
  D Variation in gas inlet arrangements

IV. Gas Bubbles
  A Rise velocity
  B Mechanism of rise
  C Merger
  D Stability
  E Maximum size
  F Bed density
  G Bypassing
  H Contact efficiency

V. Entrainment
  A Analogy to conveying
  B Transport disengaging height
  C Effective velocity

VI. Recovery Via Cyclones
  A Configuration
  B Simple theory
  C Fractional and overall efficiency
  D Loading and saltation effects
  E Attrition and bed equilibrium
  F Dipleg sizing

VII. Examples of Plant Problems
  A Grid malfunctions
  B Excessive losses
  C Internals and attrition
  D Incipient bubbling above grid
  E Scale up considerations

REGISTRATION LIMIT: 50 persons
FEE: $400 to members of AIChE, $450 to non-members. An additional $25 will be charged for on-site registration. See page 49

GENERAL INFORMATION — AIChE COURSES

EQUIREMENTS-preregistration

Registration for courses is limited and on the first-come first-served basis. Early registration is strongly recommended. If company authorization is required, do not let that deter you from registering early. You may register without company authorization or payment, however, if authorization is denied please cancel before the deadline stated below, otherwise, you will be held personally responsible for payment of the registration fee. Preregistration will be acknowledged. No cancellation will be accepted after 12:00 noon five working days prior to the scheduled date of a program, however, registrants may send substitutes by notifying AIChE National Headquarters. Applications will be accepted beyond this date provided space is available. AIChE reserves the right to cancel any program with insufficient preregistration. Refunds will be made once the attendance limit is reached. All telephone inquiries should be made to (212) 644-7526.

FEES

The fees stated in this brochure are effective January 1, 1981 through December 31, 1981. AIChE course fees are substantially lower than those offered by private educational enterprises and many university or other non-profit organizations.

The fees include all instruction, course notes and/or text material, luncheons, and coffee breaks.

All fees should be paid by the date of the course.

SPECIAL FEES

Students may register at one half the member fee, however each application must be accompanied by a letter from a faculty member indicating that the student is enrolled fulltime.

Reduced registration fee for unemployed members of AIChE are available. Full details can be obtained by contacting the AIChE Educational Services Department.

Discounts are available to organizations desiring to send five or more persons to the same program.

Reduced registration fees do not apply to the courses "Applying Programmable Calculators" and "Computer-Aided Process Design and Simulation."

HOTEL ACCOMMODATIONS

Full details on hotel accommodations will be provided all registrants in advance. Hotel reservations forms also appear on pages and of this brochure. It is strongly recommended that registrants make their hotel reservation at the earliest possible time to insure obtaining rooms at the hotel where the courses are scheduled. AIChE has reserved a block of rooms at the hotels involved. If you call the hotel to make reservations, please be sure to tell them you are attending an AIChE Continuing Education Program.

TEXTS

Most registrants will receive course notes approximately two weeks before the course is scheduled to be given. This is meant to afford attendees an opportunity to be conversant with the text prior to taking the course. We cannot stress too strongly the need for reading this material prior to attending the course.

FOOD FUNCTIONS

Registration fee for all programs includes coffee breaks at 10:00 a.m. and 3:00 p.m. each day and lunch at 12:00 noon.

CERTIFICATE OF COMPLETION & CONTINUING EDUCATION UNITS

Continuing Education Units (ceu or q) will be awarded for participation in all AIChE short courses. A Continuing Education Unit is based on 10 classroom contact hours per ceu as recommended by the National Task Force that has promulgated a "uniform unit of measurement of participation in non-credit continuing education." Thus, in most cases a participant in a one-day AIChE short course is awarded a minimum of 0.7 ceu; a participant in a two-day AIChE short course is awarded a minimum of 1.4 ceu, and a participant in a three-day AIChE course will be awarded a minimum of 2.1 ceu.

Each course participant will be provided a certificate of attendance which indicates the ceu value awarded for the short course. (Reference: Paul Grogan, "Introducing The Continuing Education Unit," Engineering Education 61, pp. 888-889, 1971).

AIChE–Registration Form

To: AIChE, Continuing Education Dept.
345 East 47th Street, New York, New York 10017

NAME ....

COMPANY ....

CITY ....

STATE .... ZIP CODE ....

BUSINESS NO. ....

SOCIAL SECURITY NO ....

- CHECK CITY*
  - ☐ Detroit
  - ☐ Montreal
  - ☐ New Orleans

*Refer to course description for cities in which each course will be offered.

- CHECK FORM OF PAYMENT
  - ☐ Please Invoice
  - ☐ Payment Enclosed
- CHECK STATUS
  - ☐ Non member
  - ☐ AIChE Member
    Member No.: ____
  - ☐ CSChE Member
    Member No.: ____

To Register For CSChE Courses See page 15

- CHECK COURSES DESIRED
  - ☐ Advanced Waste Water
  - ☐ Air Pollution Control
  - ☐ Applied Problem Solving
  - ☐ Automatic Control of Proc.
  - ☐ Catalytic Proc. in Fossil Fuel Conversion
  - ☐ Chemical Petroleum Process Fundamentals
  - ☐ Computer Aided Process Design & Simulation
  - ☐ Computer Process Control
  - ☐ Cryogenic Process Engineering
  - ☐ Design & Commercialization of Coal Conversion Processes
  - ☐ Distillation in Practice
  - ☐ Fired Heater Engineering
  - ☒ Fluid Bed Design
  - ☐ Fluid Mixing Theory & Practice
  - ☐ Fuel Alcohol Production
  - ☐ Hazard Control in Chem. Ind.
  - ☐ Managing Human Interaction
  - ☐ Managing More Productively
  - ☐ Marketing for Eng. & Sci.
  - ☐ Mathematical Modeling & Opt. for Process Design & Analysis
  - ☐ Minicomputer Systems
  - ☐ On-Line Process Measure
  - ☐ Practical Planning Methods
  - ☐ Principles of Wood Pulping
  - ☐ Process Design for Energy Conservation
  - ☐ Process Synthesis
  - ☐ Project Management
  - ☐ Solid-Liquid Separation
    Part I ☐ Part II ☐
  - ☐ Stack Sampling & Monitoring
  - ☐ Storage & Flow of Solids
  - ☐ Technical Economics
  - ☐ Thermodynamic Analysis of Improved Energy Efficiency
  - ☐ Water Quality Engineering

	For Office	Use Only	

1 2 3 4 5 6 7 8 9

Relatorio Mensal de Andamento ao CETEM

DIVISAO: DIMETE

DATA: 30 de novembro de 1981

Projeto: Estagio STI/CIDA - Tecnologia de Fornos de Leito Fluidizado
Canadian Centre for Mineral and Energy Technology - CANMET
555 Booth Street, Ottawa, ONTARIO, K1A 0G1, CANADA

Relatorio no. 02

De: Jorge L.L. Brandao,
CANMET, Room 270.

Energy, Mines and Resources Canada | Énergie, Mines et Ressources Canada

Science and Technology | Science et Technologie

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Relatorio no. 2: Novembro/81

No decorrer do mes de novembro foram realizadas as sequintes atividades:

1. Curso de tres dias em tecnologia de fornos da leito fluidizado, New Orleans, EEUU, 9-11/11/81. Relatorio de viagem em anexo.

2. Visitas tecnicas dos sequintes locais:

   i) Universidade de Toronto, Toronto, Ontario
   ii) J. Roy Gordon - Inco Research Labs, Mississauga, Ontario
   iii) Ontario Research Foundation, Mississauga, Ontario
   iv) Falconbridge Research Labs, Thornhill, Ontario

   Relatorio de viagem em anexo.

3. Planejamento para a construçao do equipamento definitivo.

4. Planejamento para obtençao de dados para o modelo em computador.

5. Continuaçao de testes com o equipamento de estudos.

6. Contatos com entidades de pesquisa relativos a modelos matemáticos em computador e equipamentos de fluidizaçao.

7. Planejamento de visitas técnicas.

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555 Booth Street,
Ottawa, Canada.
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Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada,
K1A 0G1

Programa de actividades para o proximo mes - Dezembro/1981:

1 - Continuaçao dos testes no equipamento de estudos com enfase na parte de recuperaçao do produto de arraste.

2 - Testes com partes do equipamento definitivo e compra de equipmento.

3 - Coufeccao de uma lista de dados a serem obtidos da industria para a coufecçao do modelo matematico.

4 - Visitas técnicas:

    i) New Brunswick Research and Productivity Council,
    Frederickton, New Brunswick

    ii) Gaspé Mining & Smelting (Noranda Group)
    Murdochville, Quebec

    iii) CEZ (Canadian Electrolytic Zinc, Noranda Group)
    Valleyfield, Quebec.

Ottawa, 30 de novembro de 1981

Jorge L.L. Brandao

Jorge L.L. Brandao,
eng. metalurgico

JLB/jo

TRIP REPORT

AIChE Course on Fluid Bed Reactor Design
New Orleans, LA., U.S.A.
November 9-11, 1981.

The course was divided into 7 (seven) parts, basically, as follows:

1 - Bulk solids flow
2 - Incipient fluidization
3 - Gas distribution
4 - Single particles and single bubbles; bubble flow mechanism
5 - Entrainment
6 - Recovery via Cyclone
7 - Examples of Plant Problems

NOTE: Additional details can be found in the course notes. See page 6 of this report.

1 - Bulk solids flow:

This part of the course dealt with the flow of solids (free-flowing solids) through pipes and orifices and empirical correlations were introduced to calcualte the size of holes and pipe diameters. Also discussed therein were the angle of internal friction and the angle of repose, showing their relation to incipient fluidization and solids flow in general.

2 - Incipient fluidization:

In this part of the course various empirical correlations were discussed as to their real value and practical use and it was stressed the fact that one should try not to use correlations without first having a close look at their limitations which are not always easy to determine; the best correlations seeming those which use drag coefficient versus Reynolds numbers. From this type of plot one can derive specific data more realistically.

Also discussed were terminology used in the trade such as "minimum fluidization velocity" which in reality should be called "minimum buoyancy" and the term "minimum bubbling velocity" was introduced referring to that velocity at which bubbling starts. At minimum buoyancy the pressure that the bed exerts onto the distributor equals that of the gas onto the bed. It does not imply that there are bubbles. At this point the gas is fully permeated in the bed, interstitially. After the bed is then saturated with interstitial gas, any increase in gas flow will generate bubbles in the bed. This velocity is therefore defined as minimum bubbling velocity.

3 - Gas distribution:

This part of the course was concerned with grid design and bubble formation. Correlations between pressure differentials and superficial gas velocities were discussed. A mechanism of bubble formation and growth was introduced.

Bed height was also discussed, showing its relation to maximum stable bubble diameter.

It was stressed the fact that in small beds bubbles will coalesce very fast and soon achieve the diameter of the bed promoting slug flow. It is recommended therefore that a large bed be used in order that bubbles may rise to their maximum stable diameter if scale-up correlations are desired. A reactor, as such, therefore, should be designed having in mind grid design as a first step. Jet penetration area was also investigated as well as its effect on the internals of a vessel and how it affects bed height.

Various types of distributors were shown for specific applications.

4 - Single particles and single bubbles; bubble flow mechanism:

Correlations between bubble diameter and bubble velocity were introduced and two-dimensional bed models shown in order to demonstrate bubble flow mechanism. Empirical formulae were introduced to calculate bubble velocity, bubble diameter and "cloud" diameter, as well as to calculate the volume of gas purged out of one bubble during its residence in the bed in order to determine bed height.

5 - Entrainment:

This part of the course dealt with correlations between entrainment rate and particle size distribution, gas velocity, transport disengaging height (TDH) and combinations thereof.

It was also discussed the fact that in certain applications entrainment stability may only be achieved after a long period of time (as in the case of some cracking catalysts where it may take over one year before a flow regime is attained). Sampling is then necessary in order to keep a close control of particle size in the vessel.

6 - Recovery via cyclones:

In order to better understand particle flow inside pipes, this part of the course was concerned mostly with particle conveyability through horizontal and vertical pipes. Choking velocity, pick-up and saltation velocities were discussed and correlation between these and particle size were presented. A parameter S_Λ was introduced from which one can estimate the quality of conveyability according to particle size in dilute and dense phase.

After having discussed particle conveyability and the many correlations, empirical-theoretical formulae and graphs, this last portion of the course was dedicated to cyclone design and frequent problems found in industry for which solutions were discussed.

7 - Examples of Plant Problems:

This final part of the course was dedicated to the most common problems found in industry, with regards to reactor design (mostly grid design) and cyclone design and set-up. Most problems had to do with erosion due to improper design and/or set-up.

COMMENTS

Although there was a moderate lack of response by the lecturer to questions from the audience some good practical empirical correlations and design ideas were obtained during the course. Those concerning fluid-bed design are listed below:

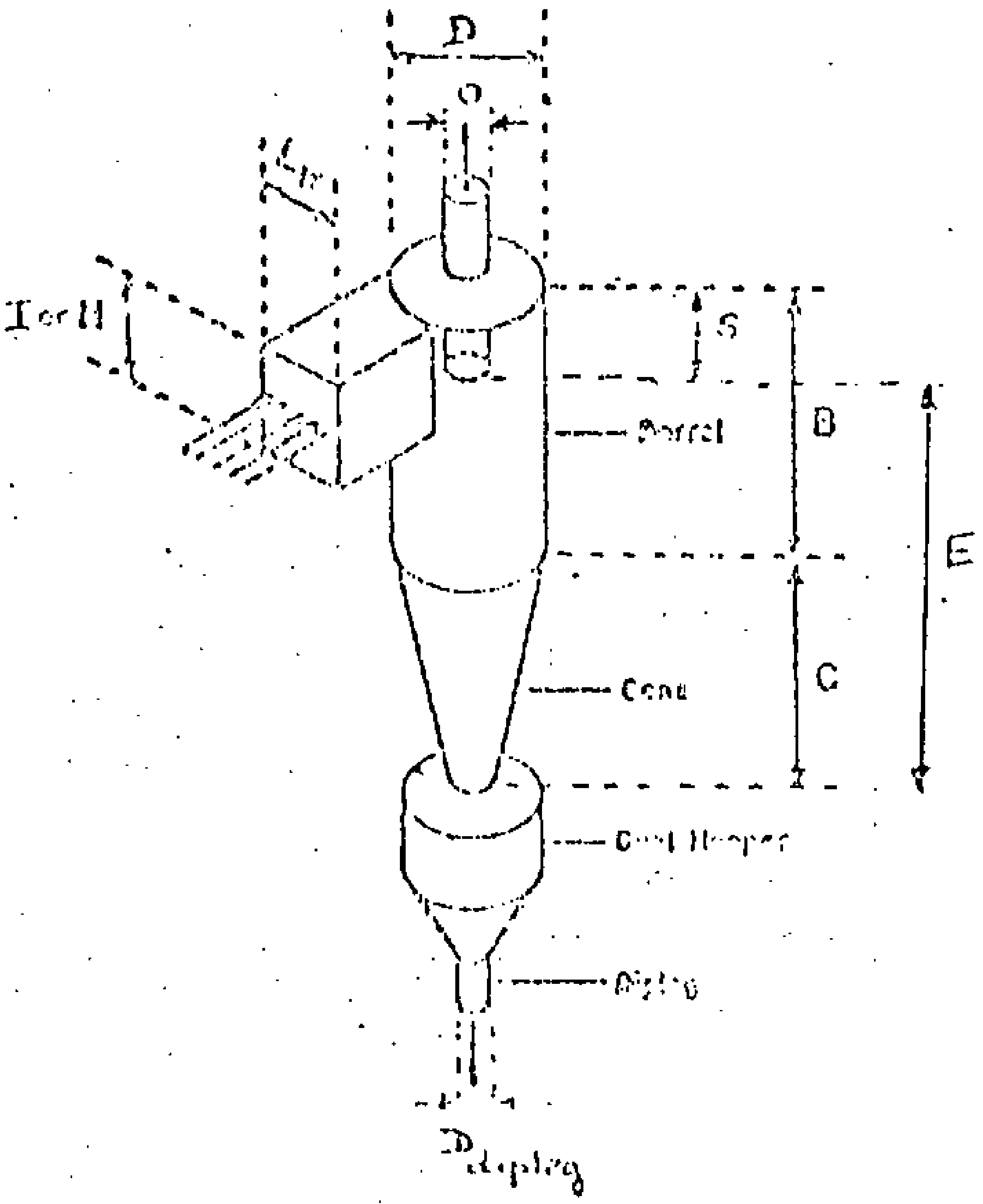
1 - Pressure taps in areas where plugging is bound to occur: pass air or a suitable gas through the pressure-tap tube and the desired pressure will be that exerted against the flow (tap-gas flow is of the order of 2 l/min).

2 - An idea for fluid bed reactor design that can provide up-scale information is the "slice-reactor". It would be a slice of a small commercial fluid-bed unit, with enough width so as to allow bubbles to rise to their maximum stable diameter. Distributor tuyeres must be as similar as possible to those in the commercial unit.

3 - Cyclone design:

A cyclone does not need to have a conical bottom. It can be simply a cylinder as long as its length is greater than 1.6 times its diameter. This can be done for small scale apparatus. In large scale units a conical bottom is preferred due to its reduced weight (less steel and refractory lining).

A typical cyclone design is illustrated below:



H = 2-3 Lw

O = 2 Lw

D = 1.6-1.7 H

S = H

B, C = 1.333 H or

E = 1.6-2 D (increases as O/I decreases)

$D_{dipleg} = 0.694 \left(\frac{W_s}{\rho_B} \right)^2$ where: Ws = solids flow

ρ_B = solids bulk density

4 - Orifice diameter: the actual orifice diameter, D, should be:
(bulk flow only) $D = D_o - 0.9\,Dp$, where: D_o = calculated orifice diameter
Dp = mean particle diameter

5 - Pressure differential correlations in fluid beds:

$$\frac{\Delta P \text{ distributor}}{\Delta P \text{ bed}} \times 100 = 30\%;\ 40\% \text{ in most cases (safety factor)}$$

$$\Delta P_{bed} = \rho_{bed} \times \text{Height of bed}$$

$$V_{orifice} = 0.8\sqrt{2\ g\ \frac{\Delta P_{dist}}{\rho f}}$$

6 - Reference material for dust collection equipment:

Emissions Control Manual
American Petroleum Institute
2101 L Street N.W.
Washington, D.C.
U.S.A. 20037

7 - Reference material for fluid beds, conveying, particle attrition, particle recovery and associated fields:

P.S.R.I. (Particulate Solid Research Inc.)
State of the Art Reports

For information write to:

Frederick A. Zenz
P.O. Box 241
Garrison, N.Y.
U.S.A. 10524

Or Call: (914) 424-3220

NOTE: P.S.R.I. is a non-profit, industry sponsored organization and most of their research is disclosed only to member companies. Government departments and agencies may not be eligible for membership.

8 - Course reference material:

1 - Text-book: Fluidization and Fluid-Particle Systems
by Zenz and Othmer
Reinhold Chemical Engineering Series

2 - AIChE course material: Fluid Bed Reactors - Design, scale-up, Problem Areas
by F.A. Zenz

Course Lecturer: Dr. Frederick A. Zenz, B.Sc., M.Ch.E., Ph.D.

November 23, 1981

Jorge L.L. Brandao
Metallurgical Engineer
CANMET
555/E Booth Street
Room 270
Ottawa, Ontario
CANADA K1A 0G1

TRIP REPORT

University of Toronto - Metallurgy & Mat. Science, Toronto, ONTARIO
Ontario Research Foundation, Mississauga, ONTARIO
Inco Metals - J. Roy Gordon Research Labs., Mississauga, ONTARIO
Falconbridge Metallurgical Labs., Thornhill, ONTARIO

1) University of Toronto: November 16, 1981 (Prof. J. Toguri)
The main purpose of this visit was to verify the existence of any projects related to fluid bed technology. They do have a long Pyrex tube, vertically mounted, where they analyse single particle reaction. They do not study to any depth fluid bed technology. They have limited knowledge on the subject.

2) Ontario Research Foundation: November 17, 1981 (Bill Fossen; Hans Brandtstatter)
At ORF there was some work done in the past on fluidized beds and some references were obtained. Since at present there are no projects dealing with fluid bed reactors the output of this visit was small and limited to reference material and confirmation of slugging in small-scale apparatus.

3) Inco Metals - J. Roy Gordon Research Labs: November 17, 1981 (Jim Eerkes)
A good deal of verbal information, some reference material and a sketch of one of the reactors used at this lab were obtained. A copy of the sketch is attached to the report.

They have two continuous operation reactors (the smaller of which is actually semi-continuous) used for roasting concentrates as well as coal and wood gasification. The discussions were related primarily to the smaller size reactor. This reactor is fed from the top and the feed is in the form of a slurry containing 70-75% solids. Most of the product is collected by the only cyclone. The remaining product is taken from the bed intermittently to maintain the pressure differential across the bed within pre-established limits. Heating is done by pre-heater, resistance tube furnace and natural gas injection in the vessel. The distributor plate was made of steel with a large number of small orifices drilled at random. The operation of the bed is controlled by a panel where pressure differential "U"-tubes are mounted and a temperature control recorder registers temperatures at various points in the vessel.

.../2.

This bed is 4 inch (10.2 cm) in diameter at the bottom and has an upper part with larger diameter - 6 inches (15.2 cm), above the bed. The reactor is wrapped with insulating material up to the top. The cyclone is not heated or cooled and discharges into a sealed container. The off-gas is then scrubbed and neutralized.

This equipment is claimed to operate well and provides some data for the larger reactor. Although it is called a fluid bed reactor due to the suspension of the particles in the vessel, it really is a slugging bed. The larger reactor is 20 cm in diameter and although running smoother than the smaller reactor it still slugs. According to the engineer responsible for its operation one would have to have a diameter of tube of at least 80 cm to obtain real fluidization. That larger 20 cm reactor has a perforated plate (similar to the smaller one) for distributor but this one lined with a layer of about 1 inch of refractory material, the holes being made through the refractory which showed cracking between the holes as well as some spalling. Some holes were blocked by coarse particles. This larger unit also has a pre-heater and instead of a tube furnace due to its larger diameter, it has a coil heater along the bed. The whole reactor is wrapped with insulating material and aluminum sheets. At present this equipment is being used for coal gasification. Its controls are similar to the smaller reactor.

4) <u>Falconbridge Metallurgical Laboratories</u>: November 18, 1981 (Ted Coulter) According to the engineer in charge of their fluid bed reactors, Inco have developed their small-scale reactors based on information obtained at this facility. Falconbridge's reactor however seem to be more elaborate than Inco's. The control panel has more resources and the overall looks of the equipment implies more expertise involved in its design. It is lined with a cast refractory, extra-light weight and a double steel wall within which 2 ring-burners at different heights, using low-pressure natural gas, provide the necessary heat to comprise with heat losses due to the relatively small size of the vessel. Prior to feeding, the reactor is pre-heated using high pressure gas through the three tuyere-caps and an extra burner higher-up in the bed. These are lit by dropping a flaming piece of paper from the open top of the tube. Once the proper temperature is achieved all internal burners are cut-off and air increased to its proper setting. Then slurry (75% solids)

.../3.

is fed from the top through a water cooled nozzle with pressure-valve alarm-control for blockage in the slurry line. A pressure tap is located in the centre of the bed, about 2 cm high form the distributor plate base, and a constant flow of air is maintained through it in order to prevent plugging. Three capped-tuyers distribute the roasting air radially with a sweeping effect on the distributor base which is refractory-lined. At the centre of the distributor base there is a depression which connects with the discharge outlet through which the reactor can be emptied. The pressure tap previously described passes through the centre of this depression. Coarser roasted particles are removed intermittently from the bed through another outlet port located higher-up with a downward angle at the wall of the reactor. This is done in order to maintain the pressure differential across the bed within pre-established limits. Roasting gases are collected at the top side of the reactor and pass through two cyclones in series, proceeding thence to the scrubber unit. The gas is neutrallized for SO_2 and SO_3, dried and filtered, and continuously analyzed for O_2.

At the first cyclone a nitrogen-purged pressure tap (about 2 l/min of N_2) is taken at its inlet line. This pressure tap is of the utmost importance. It is the lead line from which pressures differentials across the bed and across both cyclones are referred.

On the control panel one is able to monitor all natural and high pressure gas flows, air and nitrogen flows, total feed weight, pressure differentials (on magnehelic gauges), and instant bed temperature reading. A temperature recorder marks the temperatures of the ten thermocouples distributed vertically and at regular intervals in the reactor, from the tuyeres height up to the upper portion of the vessel.

Comments:

This reactor also operates on slugging, which is unavoidable in a small-scale equipment. However, if the rate of reaction is high enough at high air velocities and fine ground feed used, slugging may be lessened.

.../4.

Inco's smaller (4 in.) reactor is used as a "go-no-go" tool and little if any valuable up-scaling information is obtained. It is used to verify the chemistry of the reactions involved. At the most one can have an idea of the parameters for the larger (6 in.) reactor.

Falconbridge's reactor, by having more extensive data output may in fact provide some up-scaling parameters for an industrial size reactor.

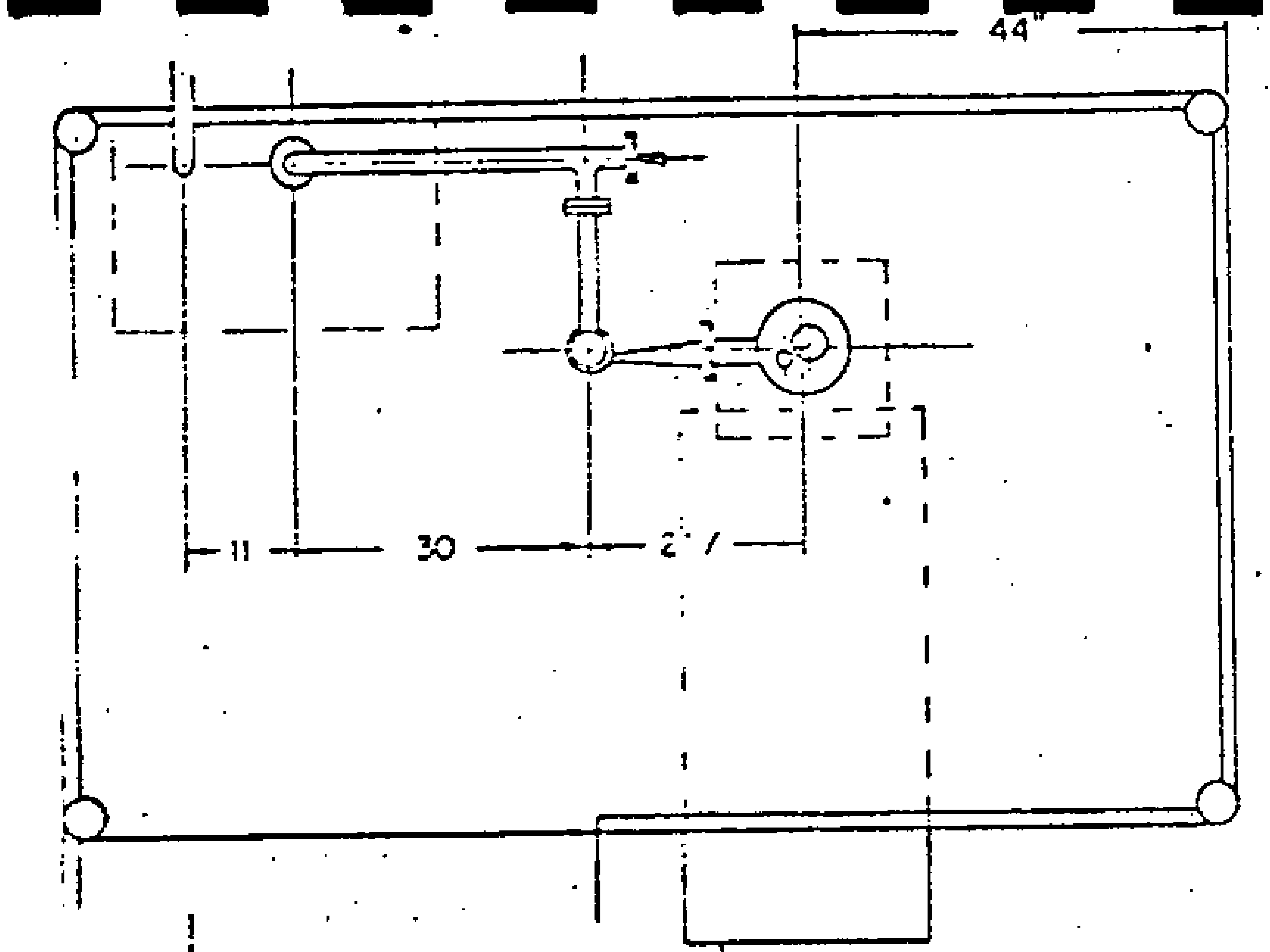
November 23, 1981

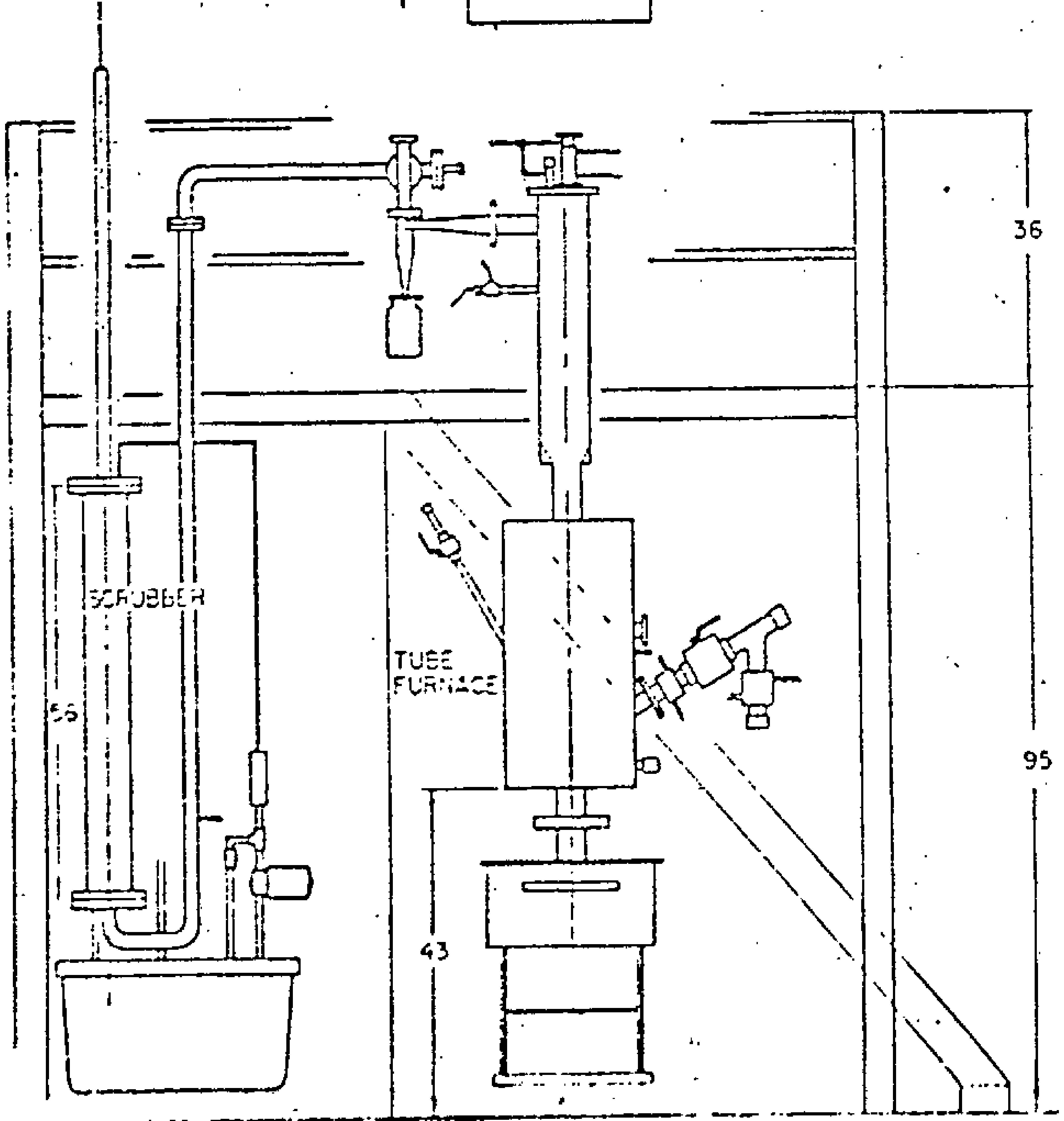
Jorge L.L. Brandao
Metallurgical Engineer
CANMET
555/E Booth Street
Room 270
Ottawa, Ontario
CANADA K1A 0G1

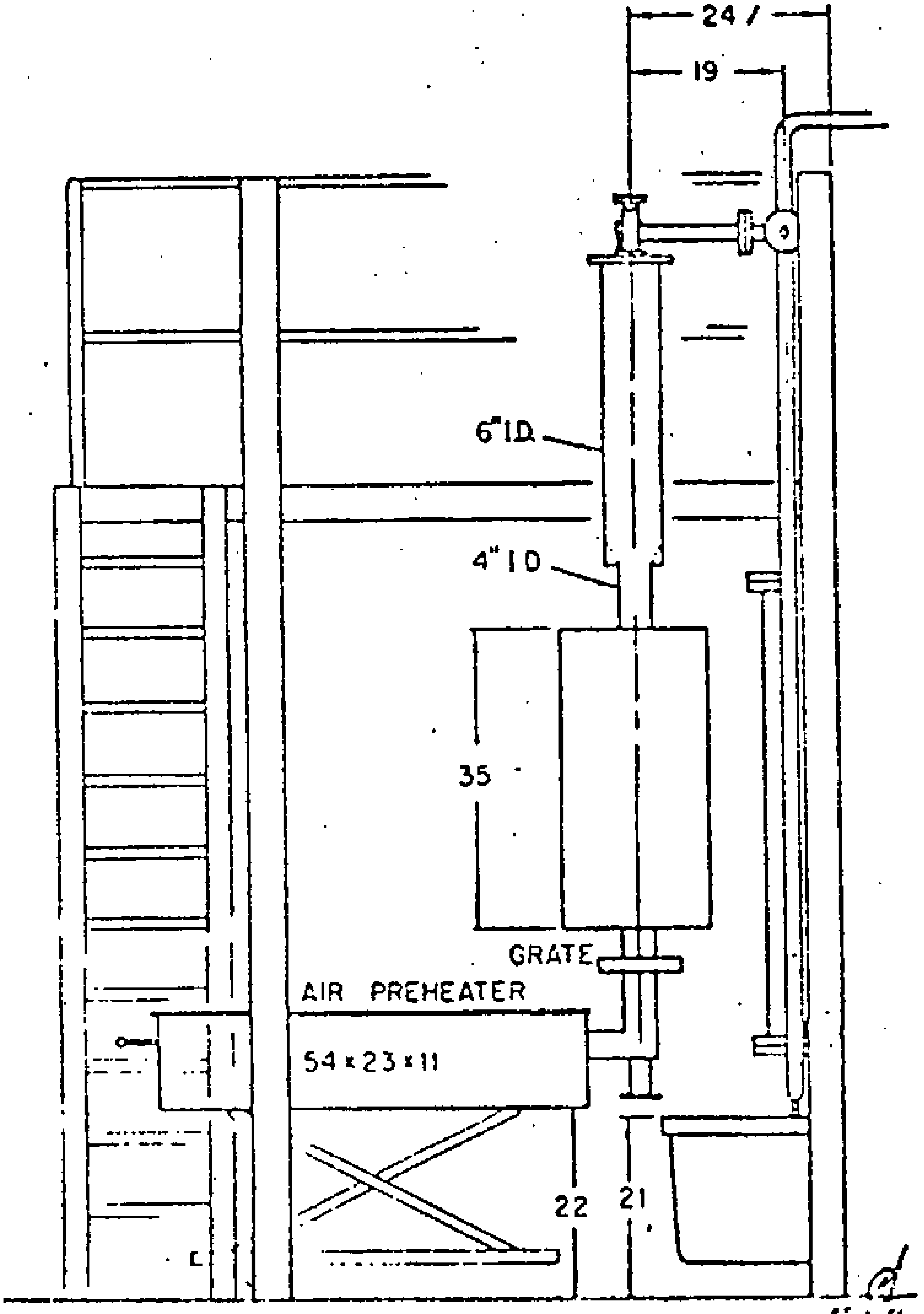
Inco

4 Inch Miniplant Fluid Bed Reactor

Scale 1/20/1" = 1"
0 5 10 20 30 Inches







Relatorio Mensal de Andamento ao CETEM

DIVISAO: DIMETE DATA: 4 de janeiro de 1982

Projeto: Estagio STI/CIDA - Tecnologia de Fornos de Leito Fluidizado
Canadian Centre for Mineral and Energy Technology - CANMET
555 Booth Street, Ottawa, ONTARIO, K1A 0G1, CANADA

Relatorio no. 03

De: Jorge L.L. Brandao,
CANMET, Room 270.

Energy, Mines and Resources Canada
Science and Technology

Énergie, Mines et Ressources Canada
Science et Technologie

Your file Votre référence

Our file Notre référence

Relatorio no. 3: Dezembro/81

No decorrer do mes de dezembro foram realizadas as seguintes atividades:

1 - Continuaçao dos testes a frio no equipamento de estudos com enfase na recuperaçao do produto de arraste (camara de descompressao, ciclones primario e secundario, recuperaçao dos super-finos), tomadas de pressao, placa distribuidora, alimentaçao. Durante estes testes foi feito uso de sucçao, procurando manter uma pressao ligeiramente negativa no reator. Esta pratica torna alimentaçao coutinua bastante simples e devera ser utilizada nos testes com o equipamento defintivo. Para tanto sera feito usa de um aspirador de pó convencional, controlando-se a sucçao por um reostato. Espera-se que parte dos super-finos possa ser recuperada no filtro do aspirador.

2 - Inicio da montagem do equipamento definitivo. Construçao de partes do reator e pre-aquecedor.

3 - Confecçao de questionario a ser levado a industria e centros de pesquisa para obtençao de um banco de dados que possibilite a confecçào de um modelo matematico em computador. Este questionario encontra-se anexo a este relatorio.

4 - Visitas tecnicas: as visitas tecnicas programmadas para dezembro tiveram de ser adiadas para janeiro, 1982.

Programa de atividades para o proximo mes - Janeiro/82:

1 - Coutinuaçao dos testes no equipamento de estudos.

2 - Coutinuaçao de testes com partes do equipamento definitivo (ciclones, forno p/reator, forno p/pre-aquecedor, reostatos, termopares, etc.).

3 - Obtencao de dados atraves do questionario para o banco de dados, durante as visitas tecnicas.

4 - Visitas tecnicas,

i) New Brunswick Research & Productivity Council,
Fredericton, New Brunswick: 11-15 Jan. 1982.

ii) Noranda Research,
Pointe Claire, Quebec: 18 Jan. 1982.

iii) CEZ (Canadian Electrolytic Zinc, Noranda Group),
Valleyfield, Quebec: 18-19 Jan. 1982.

Ottawa, 4 de janeiro de 1982

Jorge L.L. Brandao

Jorge L.L. Brandao,
eng. metalurgico.

Energy, Mines and Resources Canada
Science and Technology

Énergie, Mines et Ressources Canada
Science et Technologie

Your file Votre référence

Our file Notre référence

DATA COLLECTION QUESTIONNAIRE FOR PILOT OR COMMERCIAL FLUID BED OPERATION IN NON-FERROUS METALS PROCESSING

Company:

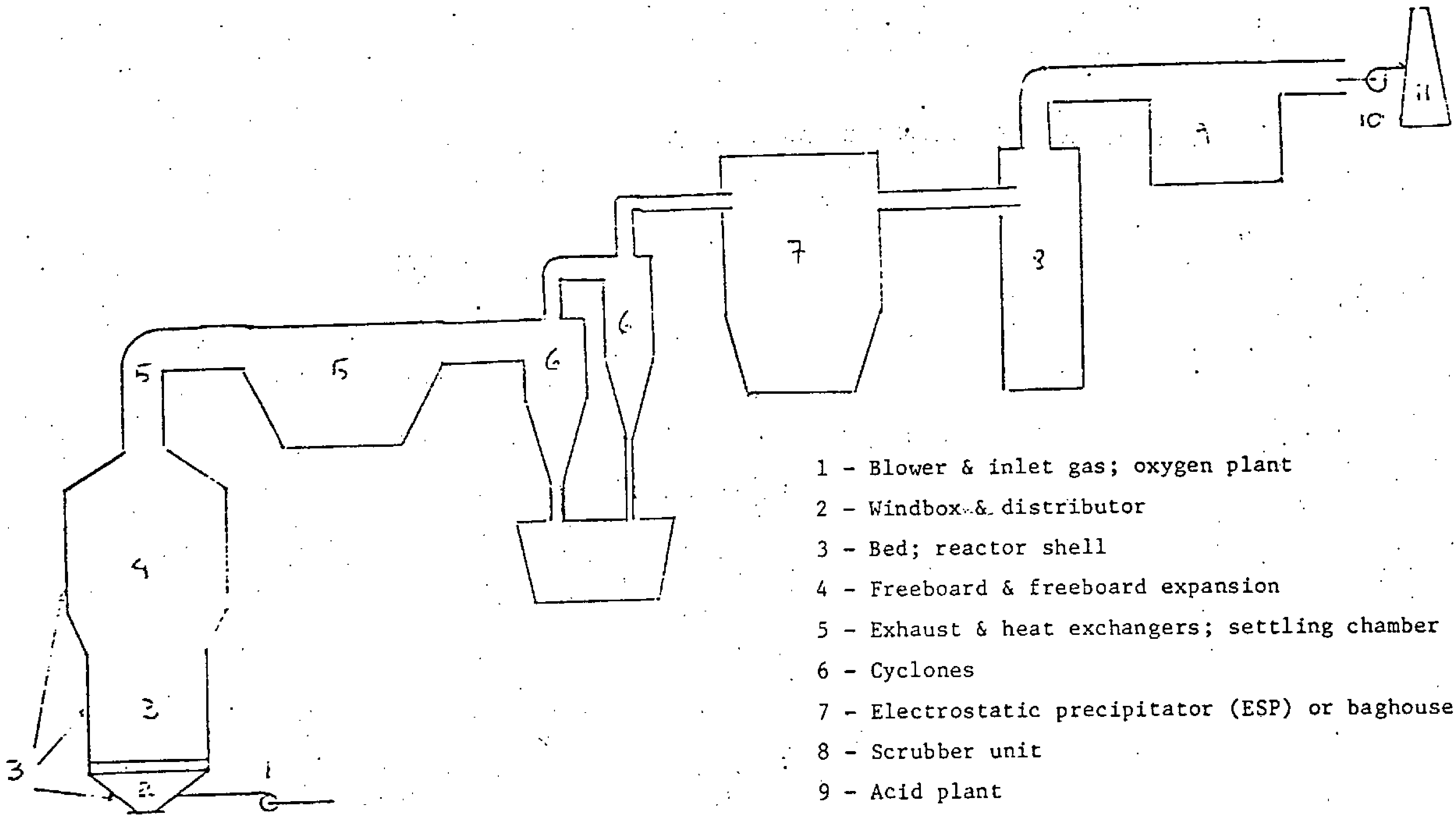
Plant Location:

Canada Centre for Mineral and Energy Technology,
555 Booth Street,
Ottawa, Canada.
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada,
K1A 0G1

Canada

Simplified Process Flowsheet with Keys to Questionnaire



1 - Blower & inlet gas; oxygen plant
2 - Windbox & distributor
3 - Bed; reactor shell
4 - Freeboard & freeboard expansion
5 - Exhaust & heat exchangers; settling chamber
6 - Cyclones
7 - Electrostatic precipitator (ESP) or baghouses
8 - Scrubber unit
9 - Acid plant
10 - Blower
11 - Stack & off-gases

1. Blower specifications:

Inlet pressures:

Pre-heating:

Location of burners:

Type of fuel:

Fuel requirements:

Excess air/O_2:

O_2 plant:

Air flow:

Temperature @ inlet (blower):

Diameter of inlet line:

OBSERVATIONS:

2. Windbox dimensions:

Refractory:

Distributor diameter & design:

No. tuyeres:

Design of tuyeres (type of steel used):

Erosion problems?:

Velocity @ tuyeres:

Refractory lining between tuyeres:

Pressure differential across distributor:

OBSERVATIONS:

3. Bed dimensions:

Depth of bed when fluidizing:

Feed rate:

Retention time:

Feed system (water cooled?):

Moisture in feed (dry or slurry):

Additives used in feed:

Discharge system (over or under flow):

Height of discharge port:

Discharge rate:

Pressure tap locations & respective readings:

Thermocouple locations (type) & readings:

Shell material:

OBSERVATIONS:

4. Temperatures across bed:

Freeboard height:

Freeboard expansion:

Refractory:

OBSERVATIONS:

5. Exit temperature of gases:

Height of outlet from distributor:

Heat exchangers (l/min):

Surface area of exchanger:

Heat removed/unit time:

Heat exchanger material (carrier? H_2O):

Water sprays? (l/min):

Pressure tap locations and readings:

Flue velocity @ inlet of settling chamber:

Flue velocity @ outlet of settling chamber:

Volume of off-gas:

Thermocouple locations & readings:

Off gas analysis:

Settling chamber dimensions:

Settling chamber:
- temperatures
- flue velocity
- pressure tap locations & readings
- refractory
- SO_2/SO_3 concentration
- Amount of product/tonne of feed (dry)
- Size analysis

OBSERVATIONS:

6. Cyclone dimensions:

No. cyclones (make):

Refractory:

Erosion problems?:

Inlet velocities & flow:

Pressure differential:

Temperature:

Efficiency:

Amount of product/tonne feed (dry):

Size analysis:

OBSERVATIONS:

7. ESP or Bag house dimensions:

No. of bags:

How frequently cleaned and replaced:

Bag material:

Acid mist formation or caking:

Size and distribution of particles:

Make:

Consumption:

Efficiency:

Temperature:

Amount dust/tonne feed (dry):

OBSERVATIONS:

ESP

Number of wires and plates

Number of sections

8. Scrubber Unit dimensions:

Scrubbing medium (wet?):

Flow rate of scrubbing medium:

Recycle? Bleed?:

Dust loading:

Discharge of effluent? Ponds?:

Nozzles type:

Packing?:

Demister:

Make:

Efficiency:

Temperature:

Gas flow (any dilution?):

OBSERVATIONS:

9. Acid plant specifications:

O_2 content:

SO_2 content/SO_3:

Tail gas analysis:

Any decolouring agent req'd (Ozone Peroxide):

Energy requirements:

Efficiency:

OBSERVATIONS:

10. Blower specifications:

Temperature of exhaust gas:

Pressure:

OBSERVATIONS:

11. Stack Dimensions:

Temperature & gas flow:

Off gas analysis:

Acid mist formation:

Burners, fuel requirements:

Refractory lining:

OBSERVATIONS:

General Information:

- Feed: Chemical & sieve analysis (particle size distribution)
- Products & by-products (settler, cyclones, ESP, scrubber, Acid Plant)
  Chemical & sieve analysis (where applicable)
- Exhaust gases: - particulate analysis
  - SO_2 concentration (any dilution?)
  - Flow rate & temperature

Additional Information

- No. Fluid Beds:
- Tonnes of dry concentrate treated per year:
- Type of concentrate:
- Downtime:
- Type of Roast (partial, dead):
- Next unit in circuit:
- Age of plant & amount of modifications since start-up:
- Process flowsheet:
- Energy balances:
- Material balances (particularly minor elements):
- Minor element distribution:
- Shell temperature surveys (beginning, middle & end of campaign):
- Velocity profiles (wherever available):
- Gas analysis:
- Wear problems & location:
- Frequent problems & others:

NOTE:

This questionnaire and all required information should be sent to the address below but NO LATER THAN MARCH 15, 1982.

Send to:

Mr. Jorge L.L. Brandao
Metallurgical Engineer
Canada Centre for Mineral &
Energy Technology
555/E Booth Street
Room 270
Ottawa, Ontario, K1A 0G1
CANADA

For further information please contact:

Dr. Peter Pint or Mr. Jorge Brandao
at (613) 995-4798.

Relatorio de Andamento à Secretaria de Tecnologia Industrial, Ministerio da Industria e Comercio: STI/MIC

Projeto: Tecnologia de Fornos de Leito Fluidizado
(estagio patrocinado pela CIDA/STI)

Estagiario: Jorge L.L. Brandao
eng. metalurgico - CETEM/CPRM/MME

Local do Estagio: Canada Centre for Mineral & Energy Technology
Energy, Mines & Resources Canada
555/E Booth Street
Room 270
Ottawa, Ontario
CANADA K1A 0G1

Data: 06 de janeiro de 1982

Program activities in the period October - December, 1981:

1. Course in fluid bed technology.
2. Fabrication of a small fluid bed reactor equipment for studies at temperatures up to 200°C.
3. Visits to research facilities.
4. Fabrication of a continuous fluid bed reactor equipment for future roasting tests.
5. Data collection questionnaire for pilot or commercial fluid bed operation in non-ferrous metals processing.
6. Forecast for program activities in the period January - March, 1982.

1. COURSE IN FLUID BED TECHNOLOGY

AIChE Today Series

Detroit, Montreal
New Orleans

FLUID BED REACTORS
ESIGN, SCALEUP, PROBLEM AREAS

Detroit, Wednesday to Friday, August 19-21, 1981

Montreal, Monday to Wednesday, October 5-7, 1981

ew Orleans, Monday to Wednesday, November 9-11, 1981

An important area of chemical engineering that has gained ncreasing prominence in recent years is fluidization technology. It is utilized in numerous industrial applications including solids transport, multiphase contracting, thermal and kinetic processes. To successfully design and operate fluidized systems, the practicing chemical engineer relies on a judicious balance of practical expèrience coupled with an understanding of the fundamentals.

This course will emphasize the basic physical phenomena and the practical approach to design in sizing fluid bed reactors; dimensioning the grid, estimating bubble size and rate of rise; calculating entrainment; choosing, locating, hanging and calculating the efficiency of cyclones and similar recovery equipment; estimating the optimum particle size and maximum bubble diameter, sizing internal heat recovery coils; estimating the size of equivalent transfer line reactors, and calculating line losses and pressure balances. Though some derivations will be reviewed they will be simple and more for background purposes to illustrate the procedure and the rationale for simplification.

The course is intended for process and design engineers, technical service groups, general "troubleshooters," plant operating group leaders and most any personnel involved in handling particulate materials especially if connected with a fluid bed plant.

COURSE OBJECTIVES

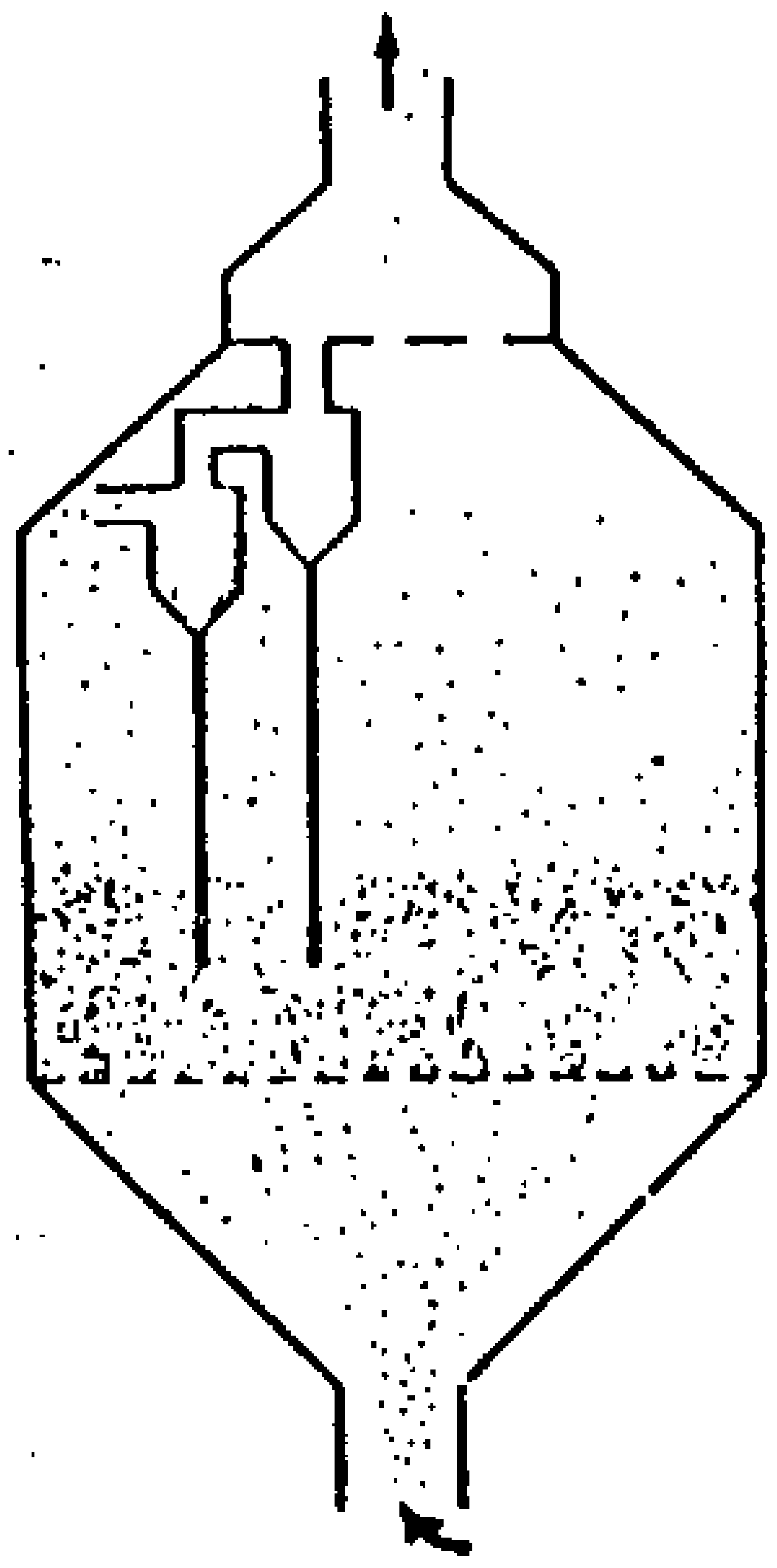
Upon completion of his course the attendees should be familiar with the background and derived relationships for calculating:

(a) Gravity, pressured, and fluidized flow rates of particle beds through standpipes and orifices.
(b) Incipient fluidization velocities, bed expansion, particle entertainment rates, and transport disengaging heights.
(c) Initial and maximum bubble sizes, merger rates, and grid characteristics.
(d) The relative degree of gas-catalyst contact to be anticipated in vessels of given dimensions.
(e) The pressure drop in dense and dilute phase pneumatic conveying.
(f) The design of cyclonic particle recovery equipment.

LECTURER

The lecturer in this AIChE Today Series will be Dr. Frederick A. Zenz, a private consultant for the past 15 years, a Professor of Chemical Engineering at Manhattan College, and Technical Director of Particulate Solid Research, Inc. a non profit industry-sponsored research company engaged in relatively large-scale experimentation in fluid beds, conveying, particle attrition, particle recovery and associated fields. He is also vice-president of Engineering at the Ducon Co., Mineola, L.I., a division of U.S. Filter Corp.

Dr. Zenz received his Bachelors degree in chemistry from Queens College, the M.Ch.E. from New York University, and the Ph.D. from what is now the Polytechnic Institute of New York. Prior to 1962 he spent a total of twenty years in industrial plant design and development with the M. W. Kellogg Co., Hydrocarbon Research Inc. and a subsidiary of Stone & Webster Engineering Corp.



COURSE OUTLINE

- **First, Second, and Third Day—9:00 A.M.-5:00 P.M.**

I. Critical Areas in Fluid Bed Design and/or Scale up
  1. Bed Solids
  2. Grid
  3. Bubbles
  4. Entrainment
  5. Recovery

II. Bed Solids
  A. Incipient fluidization
    1. Bed friction
    2. Incipient buoyancy-incipient bubbling
    3. Bed expansion and terminal velocity
    4. Correlations of incipient fluidization
    5. Empirical correlation of bed expansion
  B. Bulk solids flowability
    1. Characteristic angular properties of bulk solids
    2. Efflux rates-gravity and fluidized flow
    3. Bed support experiments

III. Grid Design
  A. Bubble formation at grid ports
  B. Gas distribution over grid areas
  C. Proximity of grid holes
  D. Variation in gas inlet arrangements

IV. Gas Bubbles
  A. Rise velocity
  B. Mechanism of rise
  C. Merger
  D. Stability
  E. Maximum size
  F. Bed density
  G. Bypassing
  H. Contact efficiency

V. Entrainment
  A. Analogy to conveying
  B. Transport disengaging height
  C. Effective velocity

VI. Recovery Via Cyclones
  A. Configuration
  B. Simple theory
  C. Fractional and overall efficiency
  D. Loading and saltation effects
  E. Attrition and bed equilibrium
  F. Dipleg sizing

VII. Examples of Plant Problems
  A. Grid malfunctions
  B. Excessive losses
  C. Internals and attrition
  D. Incipient bubbling above grid
  E. Scale-up considerations

REGISTRATION LIMIT: 50 persons
FEE: $400 to members of AIChE; $450 to non-members. An additional $25 will be charged for on-site registration. See page 49.

GENERAL INFORMATION — AIChE COURSES

REQUIREMENTS-preregistration

Registration for courses is limited and on the first-come, st-served,basis. Early registration is strongly recommended. If mpany authorization is required, do not let that deter you from registering early. You may register without company authorization or payment; however, if authorization is denied please cancel fore the deadline stated below, otherwise you will be held rsonally responsible for payment of the registration fee. Pre- gistration will be acknowledged. No cancellation will be accepted after 12:00 noon five working days prior to the scheduled date of a program; however, registrants may send substitutes by notifying IChE National Headquarters. Applications will be accepted beyond is date provided space is available. AIChE reserves the right to cancel any program with insufficient preregistration. Refunds will be made once the attendance limit is reached. All telephone inquiries should be made to (212) 644-7526

EES

The fees stated in this brochure are effective January 1, 1981 through December 31, 1981. AIChE course fees are substantially lower that those offered by private educational enterprises nd many university or other non-profit organizations.

The fees include all instruction, course notes and/or text material, luncheons, and coffee breaks.

All fees should be paid by the date of the course.

PECIAL FEES

Students may register at one half the member fee; however, ach application must be accompanied by a letter from a faculty member indicating that the student is enrolled fulltime.

Reduced registration fee for unemployed members of AIChE re available. Full details can be obtained by contacting the IChE Educational Services Department.

Discounts are available to organizations desiring to send five or more persons to the same program.

Reduced registration fees do not apply to the courses "Apply- g Programmable Calculators" and "Computer-Aided Process esign and Simulation."

HOTEL ACCOMMODATIONS

Full details on hotel accommodations will be provided all registrants in advance. Hotel reservations forms also appear on pages and of this brochure. It is strongly recommended that registrants make their hotel reservation at the earliest possible time to insure obtaining rooms at the hotel where the courses are scheduled. AIChE has reserved a block of rooms at the hotels involved. If you call the hotel to make reservations, please be sure to tell them you are attending an AIChE Continuing Education Program.

TEXTS

Most registrants will receive course notes approximately two weeks before the course is scheduled to be given. This is meant to afford attendees an opportunity to be conversant with the text prior to taking the course. We cannot stress too strongly the need for reading this material prior to attending the course.

FOOD FUNCTIONS

Registration fee for all programs includes coffee breaks at 10:00 a.m. and 3:00 p.m. each day and lunch at 12:00 noon.

CERTIFICATE OF COMPLETION & CONTINUING EDUCATION UNITS

Continuing Education Units (ceu or q) will be awarded for participation in all AIChE short courses. A Continuing Education Unit is based on 10 classroom contact hours per ceu as recommended by the National Task Force that has promulgated a "uniform unit of measurement of participation in non-credit continuing education." Thus, in most cases a participant in a one-day AIChE short course is awarded a minimum of 0.7 ceu; a participant in a two-day AIChE short course is awarded a minimum of 1.4 ceu; and a participant in a three-day AIChE course will be awarded a minimum of 2.1 ceu.

Each course participant will be provided a certificate of attendance which indicates the ceu value awarded for the short course. (Reference: Paul Grogan, "Introducing The Continuing Education Unit," Engineering Education 61, pp. 888-889, 1971).

AIChE–Registration Form

o: AIChE, Continuing Education Dept.
345 East 47th Street, New York, New York 10017

NAME ....

OMPANY ....

CITY ....

STATE .... ZIP CODE ....

USINESS NO. ....

SOCIAL SECURITY NO. ....

CHECK CITY* ☐ Montreal
☐ Detroit ☐ New Orleans

*** Refer to course description for cities in which each course will be offered.**

• CHECK FORM OF PAYMENT
☐ Please Invoice
☐ Payment Enclosed

•CHECK STATUS
☐ Non-member
☐ AICHE Member
Member No.: ____
☐ CSCHE Member
Member No.: ____

• CHECK COURSES DESIRED

- ☐ Advanced Waste Water
- ☐ Air Pollution Control
- ☐ Applied Problem Solving
- ☐ Automatic Control of Proc.
- ☐ Catalytic Proc. in Fossil Fuel Conversion
- ☐ Chemical Petroleum Process Fundamentals
- ☐ Computer-Aided Process Design & Simulation
- ☐ Computer Process Control
- ☐ Cryogenic Process Engineering
- ☐ Design & Commercialization of Coal Conversion Processes
- ☐ Distillation in Practice
- ☐ Fired Heater Engineering
- ☒ Fluid Bed Design
- ☐ Fluid Mixing Theory & Practice
- ☐ Fuel Alcohol Production
- ☐ Hazard Control in Chem. Ind.
- ☐ Managing Human Interaction
- ☐ Managing More Productively
- ☐ Marketing for Eng. & Sci.
- ☐ Mathematical Modeling & Opt. for Process Design & Analysis
- ☐ Minicomputer Systems
- ☐ On-Line Process Measure
- ☐ Practical Planning Methods
- ☐ Principles of Wood Pulping
- ☐ Process Design for Energy Conservation
- ☐ Process Synthesis
- ☐ Project Management
- ☐ Solid-Liquid Separation Part I ☐ Part II ☐
- ☐ Stack Sampling & Monitoring
- ☐ Storage & Flow of Solids
- ☐ Technical Economics
- ☐ Thermodynamic Analysis of Improved Energy Efficiency
- ☐ Water Quality Engineering

To Register For CSChE Courses See page 15

	For Office	Use Only	

123456789

TRIP REPORT

AIChE Course on Fluid Bed Reactor Design
New Orleans, LA., U.S.A.
November 9-11, 1981.

The course was divided into 7 (seven) parts, basically, as follows:

1 - Bulk solids flow
2 - Incipient fluidization
3 - Gas distribution
4 - Single particles and single bubbles; bubble flow mechanism
5 - Entrainment
6 - Recovery via Cyclone
7 - Examples of Plant Problems

NOTE: Additional details can be found in the course notes. See page 6 of this report.

1 - Bulk solids flow:

This part of the course dealt with the flow of solids (free-flowing solids) through pipes and orifices and empirical correlations were introduced to calcualte the size of holes and pipe diameters. Also discussed therein were the angle of internal friction and the angle of repose, showing their relation to incipient fluidization and solids flow in general.

2 - Incipient fluidization:

In this part of the course various empirical correlations were discussed as to their real value and practical use and it was stressed the fact that one should try not to use correlations without first having a close look at their limitations which are not always easy to determine; the best correlations seeming those which use drag coefficient versus Reynolds numbers. From this type of plot one can derive specific data more realistically.

Also discussed were terminology used in the trade such as "minimum fluidization velocity" which in reality should be called "minimum buoyancy" and the term "minimum bubbling velocity" was introduced referring to that velocity at which bubbling starts. At minimum buoyancy the pressure that the bed exerts onto the distributor equals that of the gas onto the bed. It does not imply that there are bubbles. At this point the gas is fully permeated in the bed, interstitially. After the bed is then saturated with interstitial gas, any increase in gas flow will generate bubbles in the bed. This velocity is therefore defined as minimum bubbling velocity.

3 - Gas distribution:

This part of the course was concerned with grid design and bubble formation. Correlations between pressure differentials and superficial gas velocities were discussed. A mechanism of bubble formation and growth was introduced.

Bed height was also discussed, showing its relation to maximum stable bubble diameter.

It was stressed the fact that in small beds bubbles will coalesce very fast and soon achieve the diameter of the bed promoting slug flow. It is recommended therefore that a large bed be used in order that bubbles may rise to their maximum stable diameter if scale-up correlations are desired. A reactor, as such, therefore, should be designed having in mind grid design as a first step. Jet penetration area was also investigated as well as its effect on the internals of a vessel and how it affects bed height.

Various types of distributors were shown for specific applications.

4 - Single particles and single bubbles; bubble flow mechanism:

Correlations between bubble diameter and bubble velocity were introduced and two-dimensional bed models shown in order to demonstrate bubble flow mechanism. Empirical formulae were introduced to calculate bubble velocity, bubble diameter and "cloud" diameter, as well as to calculate the volume of gas purged out of one bubble during its residence in the bed in order to determine bed height.

5 - Entrainment:

This part of the course dealt with correlations between entrainment rate and particle size distribution, gas velocity, transport disengaging height (TDH) and combinations thereof.

It was also discussed the fact that in certain applications entrainment stability may only be achieved after a long period of time (as in the case of some cracking catalysts where it may take over one year before a flow regime is attained). Sampling is then necessary in order to keep a close control of particle size in the vessel.

6 - Recovery via cyclones:

In order to better understand particle flow inside pipes, this part of the course was concerned mostly with particle conveyability through horizontal and vertical pipes. Choking velocity, pick-up and saltation velocities were discussed and correlation between these and particle size were presented. A parameter S_{Λ} was introduced from which one can estimate the quality of conveyability according to particle size in dilute and dense phase.

After having discussed particle conveyability and the many correlations, empirical-theoretical formulae and graphs, this last portion of the course was dedicated to cyclone design and frequent problems found in industry for which solutions were discussed.

7 - Examples of Plant Problems:

This final part of the course was dedicated to the most common problems found in industry, with regards to reactor design (mostly grid design) and cyclone design and set-up. Most problems had to do with erosion due to improper design and/or set-up.

COMMENTS

Although there was a moderate lack of response by the lecturer to questions from the audience some good practical empirical correlations and design ideas were obtained during the course. Those concerning fluid-bed design are listed below:

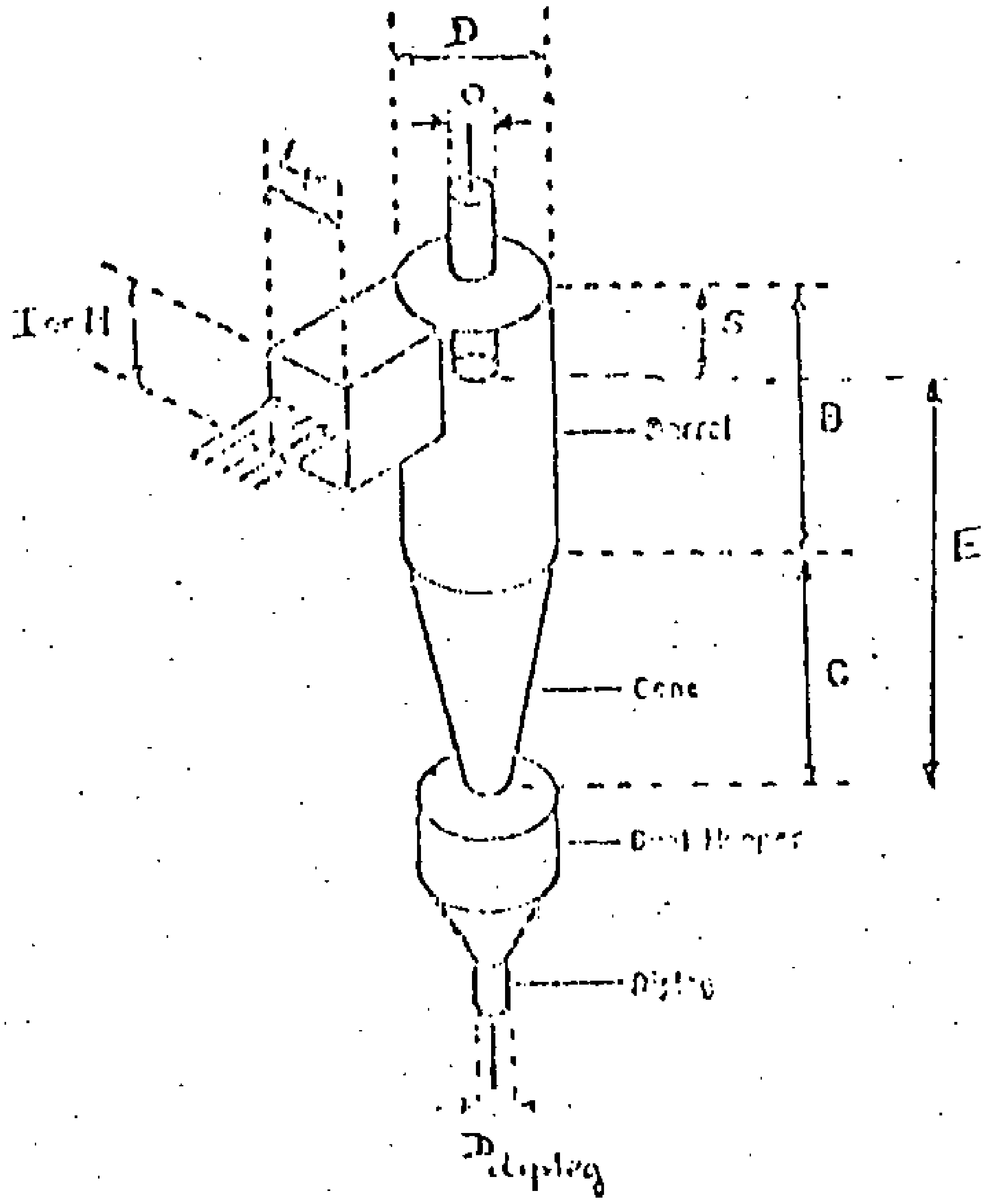
1 - Pressure taps in areas where plugging is bound to occur: pass air or a suitable gas through the pressure-tap tube and the desired pressure will be that exerted against the flow (tap-gas flow is of the order of 2 l/min).

2 - An idea for fluid bed reactor design that can provide up-scale information is the "slice-reactor". It would be a slice of a small commercial fluid-bed unit, with enough width so as to allow bubbles to rise to their maximum stable diameter. Distributor tuyeres must be as similar as possible to those in the commercial unit.

3 Cyclone design:

A cyclone does not need to have a conical bottom. It can be simply a cylinder as long as its length is greater than 1.6 times its diameter. This can be done for small scale apparatus. In large scale units a conical bottom is preferred due to its reduced weight (less steel and refractory lining).

A typical cyclone design is illustrated below:



H = 2-3 Lw
O = 2 Lw
D = 1.6-1.7 H
S = H

B, C = 1.333 H or
E = 1.6-2 D (increases as O/I decreases)
$D_{dipleg} = 0.694 \left(\frac{Ws}{\rho_B} \right)^2$ where: Ws = solids flow
ρ_B = solids bulk density

4 - Orifice diameter: (bulk flow only) the actual orifice diameter, D, should be:

$D = D_o - 0.9\ Dp$, where: D_o = calculated orifice diameter

Dp = mean particle diameter

5 - Pressure differential correlations in fluid beds:

$$\frac{\Delta P\ \text{distributor}}{\Delta P\ \text{bed}} \times 100 = 30\%;\ 40\%\ \text{in most cases (safety factor)}$$

$$\Delta P_{bed} = \rho_{bed} \times \text{Height of bed}$$

$$V_{orifice} = 0.8\sqrt{2\ g\ \frac{\Delta P_{dist}}{\rho f}}$$

6 - Reference material for dust collection equipment:

Emissions Control Manual
American Petroleum Institute
2101 L Street N.W.
Washington, D.C.
U.S.A. 20037

7 - Reference material for fluid beds, conveying, particle attrition, particle recovery and associated fields:

P.S.R.I. (Particulate Solid Research Inc.)
State of the Art Reports

For information write to:

Frederick A. Zenz
P.O. Box 241
Garrison, N.Y.
U.S.A. 10524

Or Call: (914) 424-3220

NOTE: P.S.R.I. is a non-profit, industry sponsored organization and most of their research is disclosed only to member companies. Government departments and agencies may not be eligible for membership.

8 - Course reference material:

1 - Text-book: Fluidization and Fluid-Particle Systems
by Zenz and Othmer
Reinhold Chemical Engineering Series

2 - AIChE course material: Fluid Bed Reactors - Design, scale-up, Problem Areas
by F.A. Zenz

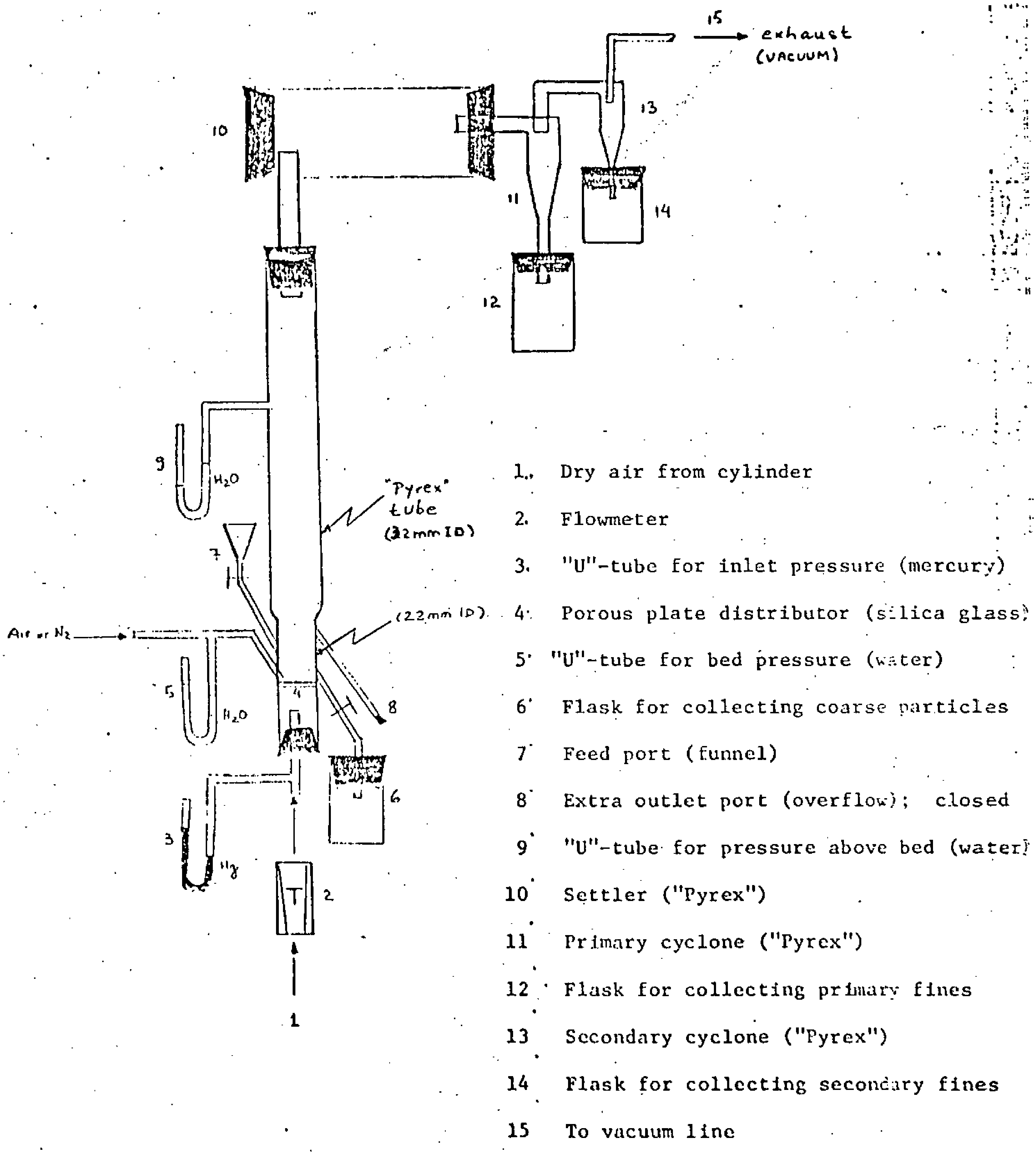
Course Lecturer: Dr. Frederick A. Zenz, B.Sc., M.Ch.E., Ph.D.

November 23, 1981

Jorge L.L. Brandao
Metallurgical Engineer
CANMET
555/E Booth Street
Room 270
Ottawa, Ontario
CANADA K1A 0G1

2. FLUID BED REACTOR EQUIPMENT FOR STUDIES AT TEMPERATURES UP TO 200°C

Sketch of the equipment:



1. Dry air from cylinder
2. Flowmeter
3. "U"-tube for inlet pressure (mercury)
4. Porous plate distributor (silica glass)
5. "U"-tube for bed pressure (water)
6. Flask for collecting coarse particles
7. Feed port (funnel)
8. Extra outlet port (overflow); closed
9. "U"-tube for pressure above bed (water)
10. Settler ("Pyrex")
11. Primary cyclone ("Pyrex")
12. Flask for collecting primary fines
13. Secondary cyclone ("Pyrex")
14. Flask for collecting secondary fines
15. To vacuum line

NOTE: Heating can be accomplished by wrapping a heating tape around the reactor tube (maximum temperature obtained: 200°C)

This equipment for studies was first built in early October. By that time it was composed only of the reactor tube in "Pyrex" and a rotameter for a maximum of 5 l/min air flow at room temperature. The top of the reactor was open to atmospheric pressure. The idea was to build a reactor for operation with minimum entrainment (carry-over of particles). The product was to be discharged at an overflow port (see no. 8 in sketch of the equipment). Since it operated at velocities of the order of 15 cm/sec, pressure at the inlet line was measured by a "U"-tube with water (low pressure). That was the only pressure tap at that time.

After the three-day course in New Orleans and visits to other research facilities (see trip reports) the earlier version of the equipment suffered a good deal of improvement. From the operational point of view it became clear that a continuous feeding reactor would have to operate on high entrainment otherwise there would be hardly any fluidization taking place at such relatively low velocities.

From Dr. Zenz's course notes and reference material a gas collecting equipment was designed and incorporated to the reactor body which itself had undergone a few changes. Two feed ports were made and later on it was decided that the top port gave better results (see no. 7 on sketch) and the lower port was then used as a pressure tap with flow of air or N_2 to avoid plugging (see no. 5). The reactor freeboard was expanded 40% (increase in diameter above the bed) to lower the rate of entrainment at high velocities. A large flow rotameter (50 l/min max. @ room temp. & press.) replaced the previous one and dry air from a compressed air cylinder was used instead of using the laboratory air line which tends to block the porous plate due to impurities in the line.

The use of vacuum was also made necessary in order to overcome blowing off at the feed port as well as to improve settler and cyclones operation. Pressure inside the reactor is now kept slightly negative and for this reason a third pressure tap was made (see no. 9). Only during actual roasting tests will operational parameters such as air flow, reactor internal pressure and feed rate, be established. Until then one can only estimate ranges within which the actual parameters will fall into.

The equipment for roasting is being assembled and the gas collection system will be exactly the same one used with the equipment for studies. While parts of that equipment are being fabricated more tests will be carried out to verify the efficiency of the settler and cyclones at various gas flows. The feed used for these tests is a zinc concentrate from Hudson Bay Mining and Smelting Ltd. For the roasting tests the zinc concentrate will come from Canadian Electrolytic Zinc Ltd. Although the suppliers are different the characteristics of both concentrates should be fairly similar. A particle size distribution will be done for this concentrate (CEZ) as it was done to the one being now used (Hudson Bay).

3. VISITS TO RESEARCH FACILITIES

TRIP REPORT

University of Toronto - Metallurgy & Mat. Science, Toronto, ONTARIO
Ontario Research Foundation, Mississauga, ONTARIO
Inco Metals - J. Roy Gordon Research Labs., Mississauga, ONTARIO
Falconbridge Metallurgical Labs., Thornhill, ONTARIO

1) University of Toronto: November 16, 1981 (Prof. J. Toguri)
The main purpose of this visit was to verify the existence of any projects related to fluid bed technology. They do have a long Pyrex tube, vertically mounted, where they analyse single particle reaction. They do not study to any depth fluid bed technology. They have limited knowledge on the subject.

2) Ontario Research Foundation: November 17, 1981 (Bill Fossen; Hans Brandtstatter)
At ORF there was some work done in the past on fluidized beds and some references were obtained. Since at present there are no projects dealing with fluid bed reactors the output of this visit was small and limited to reference material and confirmation of slugging in small-scale apparatus.

3) Inco Metals - J. Roy Gordon Research Labs: November 17, 1981 (Jim Eerkes)
A good deal of verbal information, some reference matetial and a sketch of one of the reactors used at this lab were obtained. A copy of the sketch is attached to the report.

They have two continuous operation reactors (the smaller of which is actually semi-continuous) used for roasting concentrates as well as coal and wood gasification. The discussions were related primarily to the smaller size reactor. This reactor is fed from the top and the feed is in the form of a slurry containing 70-75% solids. Most of the product is collected by the only cyclone. The remaining product is taken from the bed intermittently to maintain the pressure differential across the bed within pre-established limits. Heating is done by pre-heater, resistance tube furnace and natural gas injection in the vessel. The distributor plate was made of steel with a large number of small orifices drilled at random. The operation of the bed is controlled by a panel where pressure differencial "U"-tubes are mounted and a temperature control recorder registers temperatures at various points in the vessel.

.../2.

This bed is 4 inch (10.2 cm) in diameter at the bottom and has an upper part with larger diameter - 6 inches (15.2 cm), above the bed. The reactor is wrapped with insulating material up to the top. The cyclone is not heated or cooled and discharges into a sealed container. The off-gas is then scrubbed and neutralized.

This equipment is claimed to operate well and provides some data for the larger reactor. Although it is called a fluid bed reactor due to the suspension of the particles in the vessel, it really is a slugging bed. The larger reactor is 20 cm in diameter and although running smoother than the smaller reactor it still slugs. According to the engineer responsible for its operation one would have to have a diameter of tube of at least 80 cm to obtain real fluidization. That larger 20 cm reactor has a perforated plate (similar to the smaller one) for distributor but this one lined with a layer of about 1 inch of refractory material, the holes being made through the refractory which showed cracking between the holes as well as some spalling. Some holes were blocked by coarse particles. This larger unit also has a pre-heater and instead of a tube furnace due to its larger diameter, it has a coil heater along the bed. The whole reactor is wrapped with insulating material and aluminum sheets. At present this equipment is being used for coal gasification. Its controls are similar to the smaller reactor.

4). <u>Falconbridge Metallurgical Laboratories</u>: November 18, 1981 (Ted Coulter)
According to the engineer in charge of their fluid bed reactors, Inco have developed their small-scale reactors based on information obtained at this facility. Falconbridge's reactor however seem to be more elaborate than Inco's. The control panel has more resources and the overall looks of the equipment implies more expertise involved in its design. It is lined with a cast refractory, extra-light weight and a double steel wall within which 2 ring-burners at different heights, using low-pressure natural gas, provide the necessary heat to comprise with heat losses due to the relatively small size of the vessel. Prior to feeding, the reactor is pre-heated using high pressure gas through the three tuyere-caps and an extra burner higher-up in the bed. These are lit by dropping a flaming piece of paper from the open top of the tube. Once the proper temperature is achieved all internal burners are cut-off and air increased to its proper setting. Then slurry (75% solids)

.../3.

is fed from the top through a water cooled nozzle with pressure-valve alarm-control for blockage in the slurry line. A pressure tap is located in the centre of the bed, about 2 cm high form the distributor plate base, and a constant flow of air is maintained through it in order to prevent plugging. Three capped-tuyers distribute the roasting air radially with a sweeping effect on the distributor base which is refractory-lined. At the centre of the distributor base there is a depression which connects with the discharge outlet through which the reactor can be emptied. The pressure tap previously described passes through the centre of this depression. Coarser roasted particles are removed intermittently from the bed through another outlet port located higher-up with a downward angle at the wall of the reactor. This is done in order to maintain the pressure differential across the bed within pre-established limits. Roasting gases are collected at the top side of the reactor and pass through two cyclones in series, proceeding thence to the scrubber unit. The gas is neutrallized for SO_2 and SO_3, dried and filtered, and continuously analyzed for O_2.

At the first cyclone a nitrogen-purged pressure tap (about 2 l/min of N_2) is taken at its inlet line. This pressure tap is of the utmost importance. It is the lead line from which pressures differentials across the bed and across both cyclones are referred.

On the control panel one is able to monitor all natural and high pressure gas flows, air and nitrogen flows, total feed weight, pressure differentials (on magnehelic gauges), and instant bed temperature reading. A temperature recorder marks the temperatures of the ten thermocouples distributed vertically and at regular intervals in the reactor, from the tuyeres height up to the upper portion of the vessel.

Comments:

This reactor also operates on slugging, which is unavoidable in a small-scale equipment. However, if the rate of reaction is high enough at high air velocities and fine ground feed used, slugging may be lessened.

.../4.

Inco's smaller (4 in.) reactor is used as a "go-no-go" tool and little if any valuable up-scaling information is obtained. It is used to verify the chemistry of the reactions involved. At the most one can have an idea of the parameters for the larger (6 in.) reactor.

Falconbridge's reactor, by having more extensive data output may in fact provide some up-scaling parameters for an industrial size reactor.

November 23, 1981

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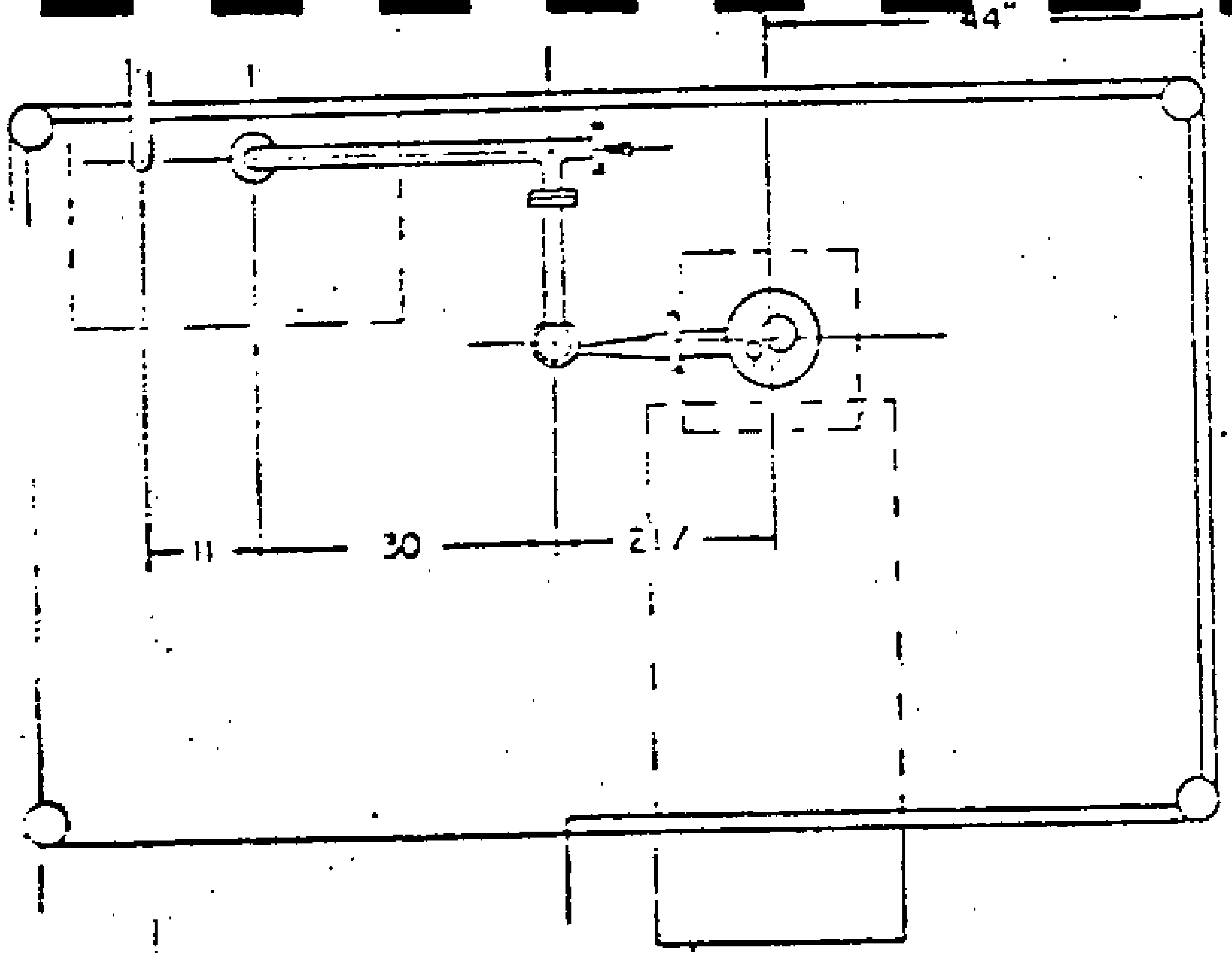
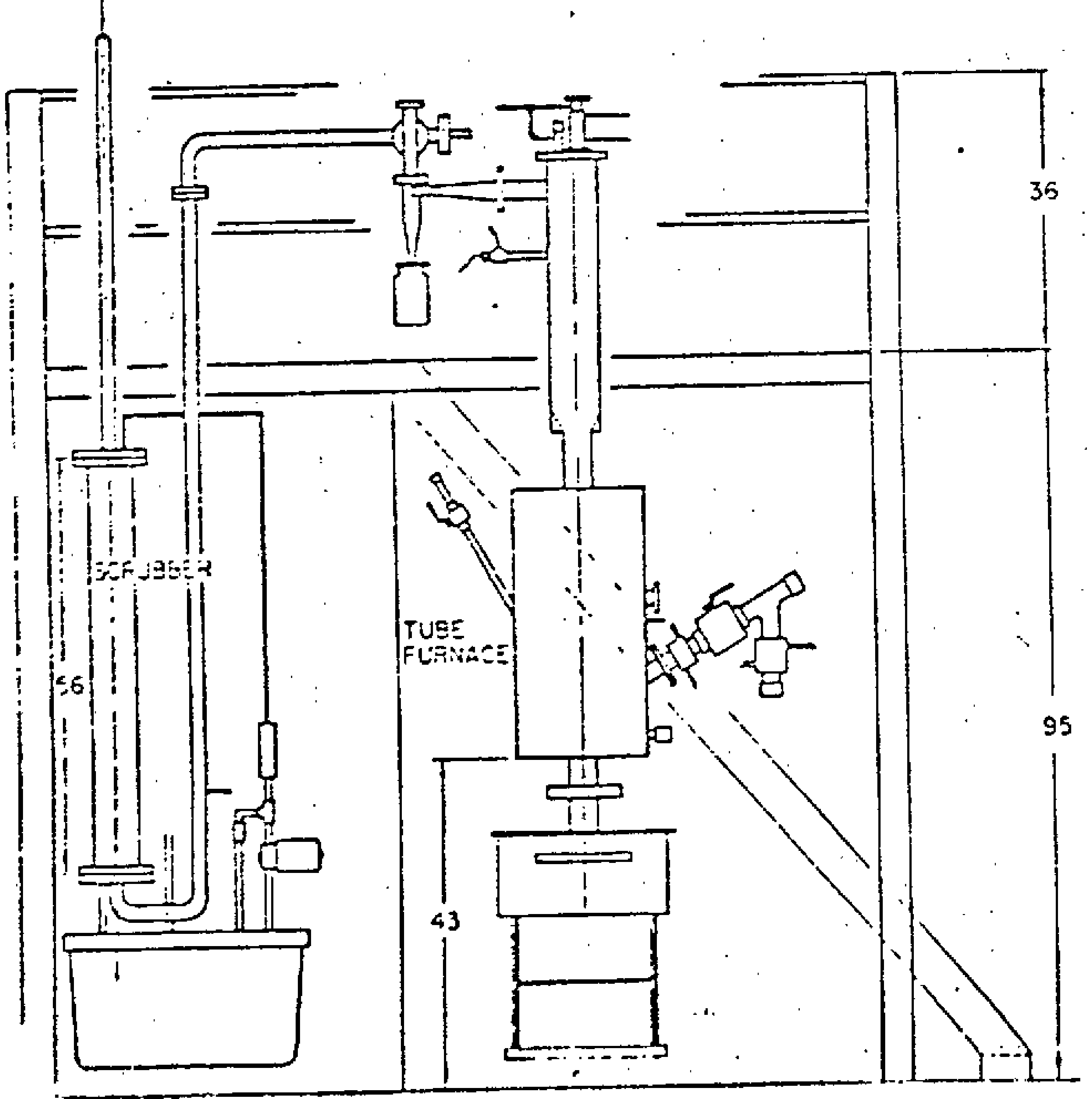
Inco

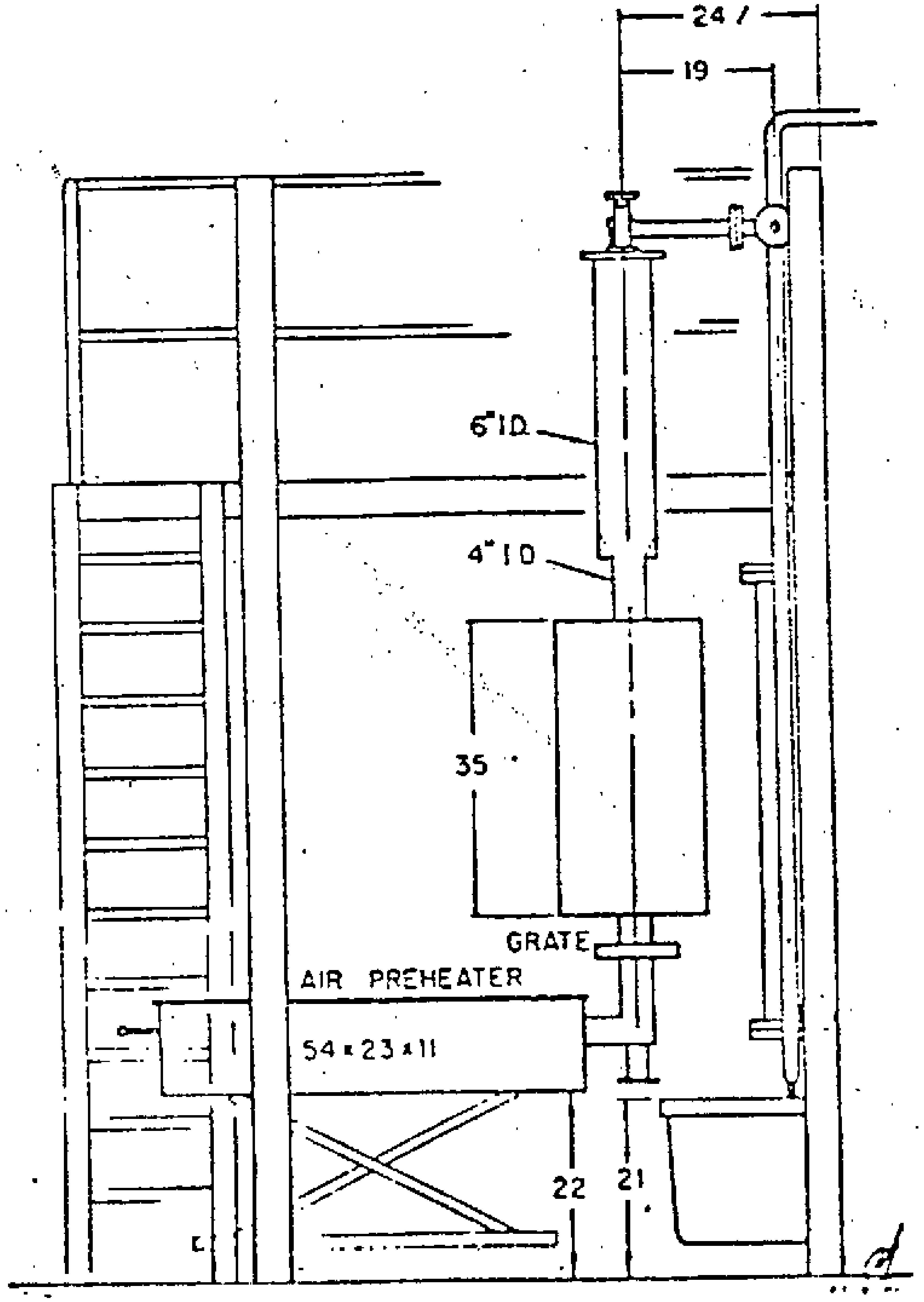
4 Inch Miniplant Fluid Bed Reactor

Scale 1/20/1" = 1"

0 5 10 20 30 Inches







4. CONTINUOUS FLUID BED REACTOR EQUIPMENT FOR ROASTING TESTS

This equipment is being assembled at present and will be similar to the one used for studies except for its larger size (60 mm I.D.). The reactor will be made out of a silica glass tube with 17% expanded freeboard. Its distributor will be made of three 316 stainless steel tuyeres with horizontal blow (eight 1.6 mm axial holes at a 30° downward angle). This has been decided after a few tests carried out with the study equipment which showed the pressure at the inlet, below the porous plate distributor, to become far too high at higher air flows (about 15 times higher than needed). Also, from previous observations porous plates produce almost no jet penetration which would indicate very poor fluidization. Coarse product will be collected at the centre of the distributor from a ½ in. tube in line with the distributor base. Feeding will be done either by a small screw-feeder or a vibrating trough or a combination of both. Pressure inside the reactor will be kept slightly negative (about - 100 mm H_2O) in order to assist feeding as well as to prevent gas leakage. Heating will be accomplished by a three-zone Lindberg "Heavy-Duty" furnace and inlet air will be pre-heated by the use of a coil tube placed inside a box furnace. The gas collecting equipment (settler, cyclones and possibly an electrostatic precipitator or bag house) will be kept at a temperature above the dew point of sulfuric acid to prevent acid mist formation. Exhaust gases will be neutralized in a scrubber with caustic solution.

Exit gases will be monitored either by gas chromatography or orsat analysis. The same procedure will apply to inlet gases in case of SO_2 and/or O_2 addition.

Operating data as well as results of roasting tests shall appear on the final report, together with a detailed sketch of the equipment.

5. DATA COLLECTION QUESTIONNAIRE FOR PILOT OR COMMERCIAL FLUID BED OPERATION IN NON-FERROUS METALS PROCESSING

Energy, Mines and
Resources Canada

Science and Technology

Énergie, Mines et
Ressources Canada

Science et Technologie

Your file Votre référence

Our file Notre référence

DATA COLLECTION QUESTIONNAIRE FOR PILOT OR COMMERCIAL FLUID BED OPERATION IN NON-FERROUS METALS PROCESSING

Company:

Plant Location:

Canada Centre for Mineral
and Energy Technology,
555 Booth Street,
Ottawa, Canada.
K1A 0G1

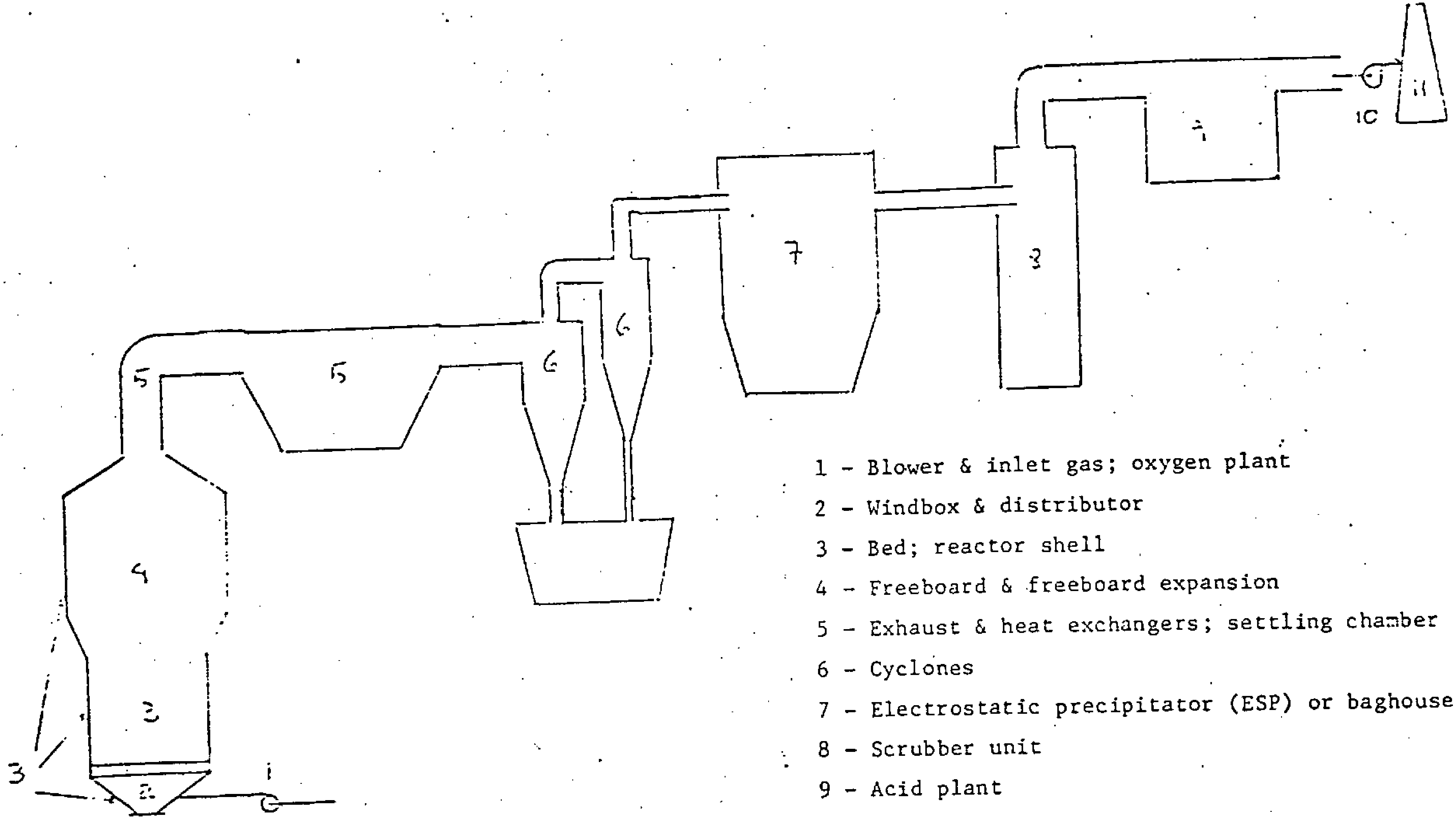
Centre canadien de la technologie
des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada,
K1A 0G1

Canadä

INTRODUCTION

The purpose of this questionnaire is to collect design and operating data on fluid bed roasters being employed by the non-ferrous metals extraction industry and their research facilities. The intention is to establish the important design and operating parameters and the interactions of the design and operating variables. The information will be used to develop a mathematical (computer) model capable of predicting behaviour of fluid bed reactors which then can be used to optimize their operation (maximize throughput, minimize downtime) and assist in designing. This will minimize reliance on the "art" of operating the reactors. All information will be confidential and will not be disclosed without the written permission of the Company.

Simplified Process Flowsheet with Keys to Questionnaire



1 - Blower & inlet gas; oxygen plant
2 - Windbox & distributor
3 - Bed; reactor shell
4 - Freeboard & freeboard expansion
5 - Exhaust & heat exchangers; settling chamber
6 - Cyclones
7 - Electrostatic precipitator (ESP) or baghouses
8 - Scrubber unit
9 - Acid plant
10 - Blower
11 - Stack & off-gases

1. Blower specifications:

Inlet pressures:

Pre-heating:

Location of burners:

Type of fuel:

Fuel requirements:

Excess air/O_2:

O_2 plant:

Air flow:

Temperature @ inlet (blower):

Diameter of inlet line:

OBSERVATIONS:

2. Windbox dimensions:

Refractory:

Distributor diameter & design:

No. tuyeres:

Design of tuyeres (type of steel used):

Erosion problems?:

Velocity @ tuyeres:

Refractory lining between tuyeres:

Pressure differential across distributor:

OBSERVATIONS:

3. Bed dimensions:

Depth of bed when fluidizing:

Feed rate:

Retention time:

Feed system (water cooled?):

Moisture in feed (dry or slurry):

Additives used in feed:

Discharge system (over or under flow):

Height of discharge port:

Discharge rate:

Pressure tap locations & respective readings:

Thermocouple locations (type) & readings:

Shell material:

OBSERVATIONS:

4. Temperatures across bed:

Freeboard height:

Freeboard expansion:

Refractory:

OBSERVATIONS:

5. Exit temperature of gases:

Height of outlet from distributor:

Heat exchangers (1/min):

Surface area of exchanger:

Heat removed/unit time:

Heat exchanger material (carrier? H_2O):

Water sprays? (1/min):

Pressure tap locations and readings:

Flue velocity @ inlet of settling chamber:

Flue velocity @ outlet of settling chamber:

Volume of off-gas:

Thermocouple locations & readings:

Off gas analysis:

Settling chamber dimensions:

Settling chamber:
- temperatures
- flue velocity
- pressure tap locations & readings
- refractory
- SO_2/SO_3 concentration
- Amount of product/tonne of feed (dry)
- Size analysis

OBSERVATIONS:

6. Cyclone dimensions:

No. cyclones (make):

Refractory:

Erosion problems?:

Inlet velocities & flow:

Pressure differential:

Temperature:

Efficiency:

Amount of product/tonne feed (dry):

Size analysis:

OBSERVATIONS:

7. ESP or Bag house dimensions:

No. of bags:

How frequently cleaned and replaced:

Bag material:

Acid mist formation or caking:

Size and distribution of particles:

Make:

Consumption:

Efficiency:

Temperature:

Amount dust/tonne feed (dry):

OBSERVATIONS:

ESP

Number of wires and plates

Number of sections

8. Scrubber Unit dimensions:

Scrubbing medium (wet?):

Flow rate of scrubbing medium:

Recycle? Bleed?:

Dust loading:

Discharge of effluent? Ponds?:

Nozzles type:

Packing?:

Demister:

Make:

Efficiency:

Temperature:

Gas flow (any dilution?):

OBSERVATIONS:

9. Acid plant specifications:

O_2 content:

SO_2 content/SO_3:

Tail gas analysis:

Any decolouring agent req'd (Ozone Peroxide):

Energy requirements:

Efficiency:

OBSERVATIONS:

10. Blower specifications:

Temperature of exhaust gas:

Pressure:

OBSERVATIONS:

11. Stack Dimensions:

Temperature & gas flow:

Off gas analysis:

Acid mist formation:

Burners, fuel requirements:

Refractory lining:

OBSERVATIONS:

General Information:

- Feed: Chemical & sieve analysis (particle size distribution)
- Products & by-products (settler, cyclones, ESP, scrubber, Acid Plant)
  Chemical & sieve analysis (where applicable)
- Exhaust gases:
  - particulate analysis
  - SO_2 concentration (any dilution?)
  - Flow rate & temperature

Additional Information

- No. Fluid Beds:
- Tonnes of dry concentrate treated per year:
- Type of concentrate:
- Downtime:
- Type of Roast (partial, dead):
- Next unit in circuit:
- Age of plant & amount of modifications since start-up:
- Process flowsheet:
- Energy balances:
- Material balances (particularly minor elements):
- Minor element distribution:
- Shell temperature surveys (beginning, middle & end of campaign):
- Velocity profiles (wherever available):
- Gas analysis:
- Wear problems & location:
- Frequent problems & others:

NOTE:

This questionnaire and all required information should be sent to the address below but NO LATER THAN MARCH 15, 1982.

Send to:

Mr. Jorge L.L. Brandao
Metallurgical Engineer
Canada Centre for Mineral & Energy Technology
555/E Booth Street
Room 270
Ottawa, Ontario, K1A 0G1
CANADA

For further information please contact:

Dr. Peter Pint or Mr. Jorge Brandao at (613) 995-4798.

6. FORECAST FOR PROGRAM ACTIVITIES IN THE PERIOD JANUARY - MARCH, 1982

For the next three-month period the following activities are outlined:

1. Visits to pilot and industrial fluid bed roasting to collect data for the mathematical model as well as to verify the various modes of operation used by industry and research facilities.

   Three of such visits are scheduled for the month of January, 1982, as follows:

1.	Jan. 11-15, 1982:	New Brunswick Research & Productivity Council, Fredericton, New Brunswick.
2.	Jan. 19, 1982:	Noranda Research, Pointe Claire, Quebec.
3.	Jan. 19-20, 1982:	Canadian Electrolytic Zinc (CEZ) Valleyfield, Quebec.

   Other visits will be scheduled for the following months.

2. Preparation of computer program and compilation of data collected from industry and research facilities.

3. Roasting tests: it is estimated that of the order of 20 such tests will be carried out until the end of March, 1982. Results obtained thereof will be included in the final report.

Ottawa, January 6th, 1982

Jorge L.L. Brandao

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CANADA K1A 0G1

Relatorio Mensal de Andamento ao CETEM

DIVISAO: DIMETE DATA: 2 de fevereiro de 1982

Projeto: Estagio STI/CIDA - Tecnologia de Fornos de Leito Fluidizado
Canadian Centre for Mineral and Energy Technology - CANMET
555 Booth Street, Ottawa, ONTARIO, K1A OG1, CANADA

Relatorio no. 04

De: Jorge L.L. Brandao,
CANMET, Room 270.

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Science and Technology
Science et Technologie

Your file Votre référence

Our file Notre référence

Relatorio no. 4: Janeiro/82

No decorrer do mes de janeiro foram realizadas as seguintes actividades:

1 - Continuaçao de testes no equipamento de estudos com enfase na parte de alimentaçao continua, eficiencia da camara de descompressao e ciclones. Foi instalada uma coluna de escrubagem para observar a eficiencia do equipamento de recuperaçao do produto de arraste. Foi instalado também um compressor para ser utilizado como bomba de vacuo e os resultados foram positivos.

2 - Continuaçuo de testes com o equipamento definitivo:
- Tubeiras: verificando eficiencia e qualquer ajuste necessario
- Compressor (vacuo)
- Alimentaçao
- Recuperaçao dos produtos de arraste e "underflow".
- Rotametros
- Tubos em "U" para pressoes em diversos pontos do equipmento
- "Windbox", ponto de junçao da linha de pre-aquecimento e tubeiras (tomadas de pressao e temperatura)

3 - Reuniao com Daniel Laguitton (especialista em simulaçao por computador) para determinar as possibilidades de elaborar-se um modelo matematico para fornos de leito fluidizado na industria dos nao-ferrosos.

O resultado desta reuniao foi positivo embora seja necessario amplo interesse da industria para levar a cabo tal objetivo.

4 - Visitas tecnicas:

(i) New Brunswick Research & Productivity Council (R.P.C.)
Fredericton, New Brunswick: 11-15 Jan., 1982

(ii) CEZ (Noranda Group)
Valleyfield, Quebec: 22 Jan., 1982

NOTA: Ver relatorios de visita em anexo ("Trip Reports").

Canada Centre for Mineral and Energy Technology,
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January 27, 1982

TRIP REPORT

New Brunswick Research and Productivity Council - (R.P.C.)
Fredericton, New Brunswick.

Canadian Electrolytic Zinc - CEZ (Noranda Group),
Valleyfield, Quebec.

i) R.P.C.: January 11-15, 1982 (Roy Boorman, Bob Salter, Ross Gilders)

The main purpose of this visit was to observe the operation of RPC's 6-inch diameter fluid bed reactor, as well as to obtain as much information as possible on their 14-in. diameter reactor as well as RPC's sulphation roasting process. Included in this report are a drawing of their 14. in. reactor and a description of their sulphation roasting process including actual test data, a flowsheet of the process and other pertinent technical information.

During my five-day visit to RPC I was in direct contact with Ross Gilders who is the engineer responsible for the operation of their fluid bed reactors. Only the 6. in. reactor was being used then, as part of a project consisting of desulphurizing effluent gases during the combustion of high-sulphur coals with the use of oil shales. The purpose being to determine the best ratio of coal/oil shale for various types of shales, the efficiency of the reaction and to analyse calcine products formed by the reaction of SO_2 from the coal with carbonates in the shale.

It was most interesting to find out the origin of their 6 in. reactor. RPC bought it from "Hazen" over 9 years ago. The original equipment, according to Bob Salter (Senior Process Chemist), never worked, and they had to make extensive modifications. To-day, all that is left of the original equipment is the 316 stainless steel reactor tube and the furnace shell, the core of which was modified in order to serve as support of the modified reactor heating system. Their distributor plate, made of stainless stell, has several "mushroom" tuyeres, placed at random. The reactor is fed through

.../2.

a tube at bed height and the feed is "blown-in" since the reactor has a positive pressure. There is no suction between the reactor and the venturi-scrubber. This practice generates several problems, such as: low cyclone efficiency, large air 'infiltration' which is used to blow in the feed, gas leaks, blockages in the feed inlet and in the venturi-scrubber. It was noticed that there is a lack of pressure taps which would be of great necessity for such pressurized system. This reactor as a whole seems to lack in control mechanisms but seemed to be efficient enough for their small-scale continuous tests.

Effluent gases are analysed every 18 minutes by a gas chromatograph which pulls the same amount of sample everytime according to a pre-set program and records on a chart as peaks which are compared visually with "standard peaks" and later on analysed more thoroughly. This technique seemed to be quite efficient and there were no problems in that area. Drager tubes were used for very low SO_2, which would not appear on the chromatogram.

The heating system used for the reactor, pre-heater and gas collecting system consists of standard shielded electrical resistances which are wrapped around the surface to be heated, in one or more layers. A thermocouple is placed touching each series of elements in order to control their maximum operating temperature (800°C for those elements). This system seems to be very effective and no problems were reported. The heating elements were said to last up to three years, if kept at 800°C, or whatever their maximum operating temperature may be.

For coal combustion the fines collected in both cyclones are continuously re-cycled to the reactor by star-feeders and temperature is kept constant by a water coil located on top of the distributor plate which has water inlet and outlet tubes passing through it and connected to the coils by swage-locks.

The weights of the materials being fed are calculated by the weight difference of the bins. For the oil shale an ordinary scale with weights is used whereas for the coal the entire feed system is continuously weighed by tour strain gauges located at each corner of the support giving continuous mV readings that are calibrated to grams, appearing on a digital read-out monitor. Water to the coil and air of reaction and feed lines are controlled by rotameters.

ii) C.E.Z. January 22, 1982 (Philippe Krick, Pierre Duhaime)

After a briefing with Philippe Krick, Technical Superintendent, when we, Peter Pint and myself, explained the purpose of our visit to CEZ and discussed on various aspects of zinc sulphide roasting practice and exchanged a few ideas, we introduced our "Data Collection Questionnaire" for which we received a very positive reaction and a promise to send it back to us with all required information. Pierre Duhaime, plant engineer, who was also present, explained that he had done some data collection in the past, as a co-op student and had produced a simplified mathematical (computer) model for the roasting reactors at CEZ. This model, however, was crude and some of its data are now outdated.

Pierre Duhaime guided us on a visit of the plant, starting at the unloading of concentrates, number 1 and 2 reactors and their larger reactor, number 3. We entered the control room where the entire roasting and gas collecting system is monitored. We then proceeded to the leaching and purification sections and finally to the zinc electrowinning area where we observed the manual stripping of zinc cathodes and proceeded to the newer part of the electrowinning area where wider cathodes are automatically stripped. We could not observe this operation for the "MITSUI" automatic stripper was out of service. From there we passed by the zinc moulding area where zinc cathodes are molten in electric furnaces, mixed with alloying elements according to customer specification, and poured into top-heated moulds where zinc ingots are cast and piled for inspection and stripping.

We did not visit the acid plant and were explained that we could not visit their mercury scrubber due to a licensing agreement between CEZ and the scrubber manufacturer (Boliden-Norzink).

CEZ Roasting Plant: Dead Roast

The roasting plant is composed of three Lurgi-type fluid bed roasters. The first two, with 32m^2 bed area (200 TPD each), were built in 1967 and the third one, having more than twice the bed area of the previous two, 72m^2 (450 TPD), went in operation in 1975. A fourth reactor is under construction and will be similar to the larger one, with 76m^2 bed area. Both large reactors have two side-by-side slinger-feeders whereas the smaller reactors need only one each. The feed is a blend of zinc-concentrates (about 52% Zn, 33% S, 11% Fe, 1.0% Pb; see Table 1 in the Duhaime-Krick paper, annexed). Temperature in the reactor stays around 950°C and is controlled by adding water to the feed, injecting water through the feed-port and by water-coils. A good portion of the steam used in the plant is produced at the roasters. The feed has 8% moisture and is 92% below 200 mesh. The calcine is finer, 95% below 325 mesh, and less dense. Superficial gas velocity is about 80 cm/sec and pressure below the distributor is about 1300 mm H_2O for the smaller reactors and about 1500 mm H_2O for the larger.

.../4.

The tuyeres are of the constricted-tube type, 3/16 in. diameter at the top inch or so, and about 1/2 in. diameter through to the bottom part and measures about 6 inches in length. This is done to give it enough mechanical resistance as well as lower pressure drop. Air temperature is about -5°C in the winter and about 20°C in the summer. There is no pre-heating of reacting air. There are 100 tuyeres per m^2 of distributor area, for all three reactors.

About 5% of the calcine product in collected by overflow, continuously, and the remainder entrained product is collected 25% in the settler (heat exchanger), 30% in each of the two cyclones and the rest in the electrostatic precipitators, which have an efficiency of 98.8% (Joy and Wheelabrator). The third reactor has a "Flakt" ESP with a lower efficiency, 98% which causes problems for the acid plant. The fourth reactor will have a "Wheelabrator" ESP which has proven to give the best overall performance.

It is assumed that the overflow calcine has an average residence time of 5 hours whereas the entrained calcine product has only 2 hours. There is less than 2% sulphides in the calcine products, which are composed mostly of zinc oxide and zinc ferrite. The latter is recovered by jarosite precipitation. Reactor effluent gas has 11-14% SO_2 and 5-6% O_2 as well as other constituents (N_2, some CO/CO_2, etc....). The effluent is diluted naturally to about 7% SO_2 for admission into the acid plant circuit.

The annual throughput of the plant is approximately 220,000 tonnes of dry concentrate. About 1/3 of the total amount of concentrate received at CEZ is roasted at Allied Co., nearby, which have an old fluid bed reactor, straight section (i.e., no expanded freeboard) and operate at 850°C, one hundred degrees lower. Their calcines have less zinc in the ferrites and it is suspected that it is due to the characteristics of the concentrate they treat, which is somewhat different from the blend CEZ use.

Attached to this report are general flowsheets of the plant (roasting, leaching, purification, electrolysis, and casting) and an article put together by Pierre Duhaime and Philippe Krick on the Fluid-Bed Roasting Operation of Zinc Minerals. This article has some errors and should be read carefully. In the overall it is a very good piece of information that should be part of the reference material of anyone who is involved in the field of fluid bed technology.

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ANNEX 1

New Brunswick Research and Productivity Council

APPENDIX A1

RPC SULPHATION ROAST-LEACH-ELECTROWINNING PROCESS DEVELOPMENT PROGRAM

A1.1 DESCRIPTION OF SEMI-PILOT PLANT

The roasting capacity of the plant was approximately 120-150 kg feed per day, on a three shift basis. Actual operation was limited to two consecutive shifts per day. The roasting equipment is shown in Plate 1 and illustrated schematically in Figure A1-1. The fluidized reactor, which was constructed of 316 stainless steel, measures 30 cm (14 in.) in diameter by 120 cm (47 in.) in height with an adjustable fluidized bed height of 40-55 cm. The gas distributor at the base of the reactor consisted of 19 mushroom-type tuyeres, each with six, 1 mm diameter holes and a total pressure drop across the distributor of 45 cm H_2O. The gas cleaning system includes a 20.3 cm (8 in.) diameter primary cyclone in series with a 15.2 cm (6 in.) diameter secondary cyclone and a dry single electrode electrostatic precipitator (cottrell).

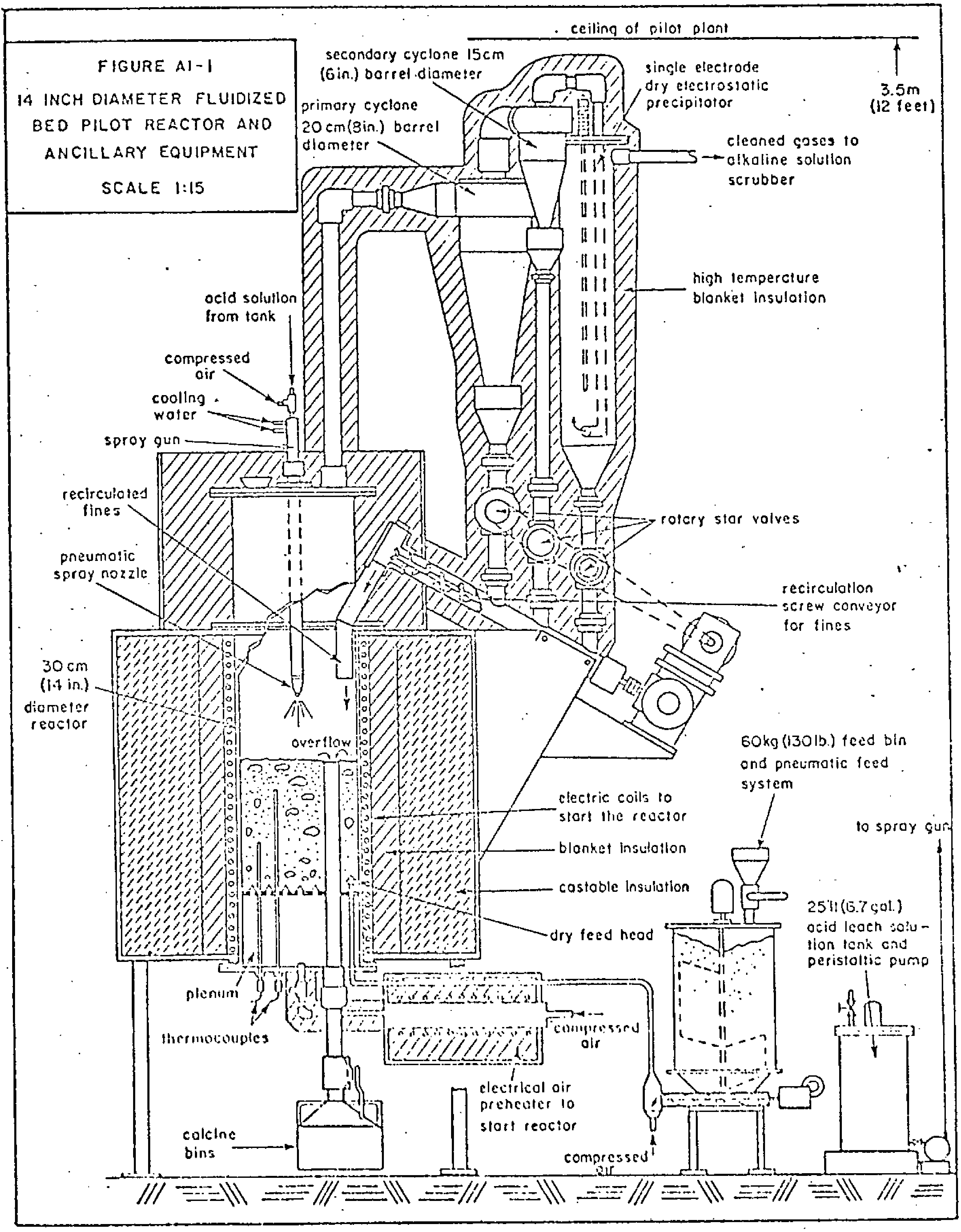
Dust collected in the gas cleaning system discharged through individual rotary star valves rotating at 45 and 35 RPM and was continuously recirculated back into the reactor by means of a 3-inch diameter slanted screw feeder rotating at 25 RPM, as shown in Figure A1-1.

Slurry feed was sprayed from the top of the reactor by means of a compressed air operated gun, cooled with a

FIGURE A1-1

14 INCH DIAMETER FLUIDIZED BED PILOT REACTOR AND ANCILLARY EQUIPMENT

SCALE 1:15



water jacket. Provision was made for varying the distance of the gun nozzle from the fluidized bed surface. Calibration data for this feed system is given in Figure A6-1. Dry feed was measured to the pneumatic injection system by means of a screw feeder, while slurry was pumped to the gun with a variable speed peristaltic rotary pump. The recirculated hot acid leach filtrate was sprayed into the reactor from the top, using the same gun and pump used for slurry feeding.

The reactor was started by means of electric heating coils wrapped around its body and with an electric air preheater. During operation, the unit was autothermally maintained, with no external power normally required.

External power, sufficient to compensate for the relatively high conductive heat losses associated with a reactor of this comparatively small size, was required to maintain thermal balance in the interlocked trials where hot acid leach filtrate was decomposed in the reactor.

The hydrometallurgical section of the RPC semi-pilot plant is illustrated in Figure A1-2. Design capacity was sufficient to handle 150 kg calcine per day on a three shift basis, however, actual operation was adjusted according to daily roaster production. Each calcine was leached and treated in each unit as a batch due to equipment and manpower limitations. Several operations were internally fully continuous such as copper solvent extraction and zinc electtrowinning and the leaching operations were semi-continuous

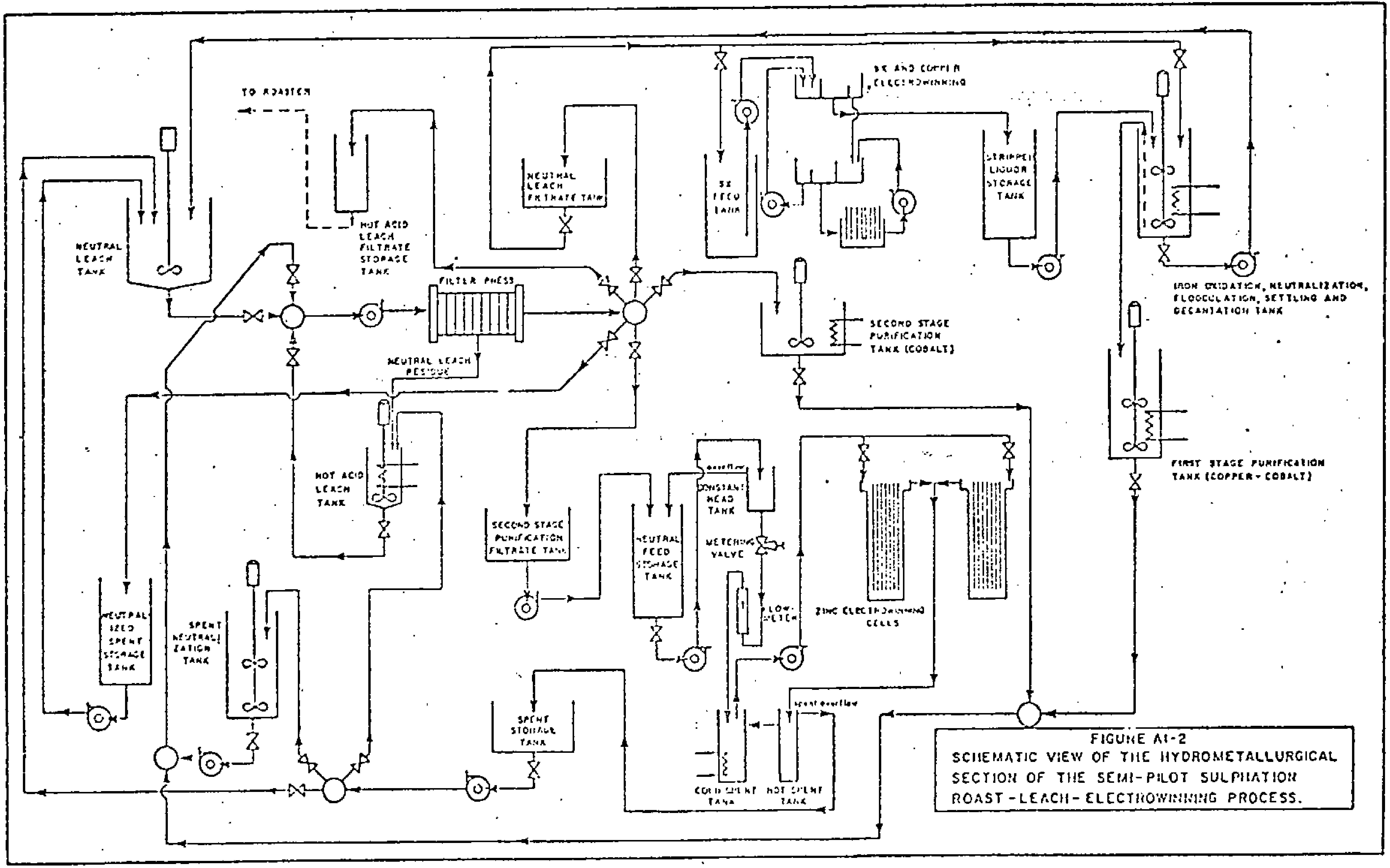


FIGURE A1-2
SCHEMATIC VIEW OF THE HYDROMETALLURGICAL SECTION OF THE SEMI-PILOT SULPHATION ROAST-LEACH-ELECTROWINNING PROCESS.

in that acidity was kept constant by continuous addition of acid. Plate 2 shows typical procedures during operation. Detailed discussion of equipment and operation is given in Section A1.3.

A1.2 ROASTING SECTION

121 Feeds Tested

Five different products were tested in the roasting circuit. Three products are representative of low grade zinc concentrates with zinc contents from 22 to 32% and varying amounts of lead and copper. Also tested was a 51% zinc concentrate and a blend of a tailings refloat (30% Zn) product and a ferrite residue from a conventional dead roast zinc plant.

The ranges of chemical compositions of the samples tested are given in Table A1-1. The detailed chemical analyses of feeds for each run are given in Table A1-2.

For the slurry feed tests, feed with approximately 10% moisture was slurried with spent electrolyte. For the dry feed tests the sulphide material, which was originally milled to 85% minus 325 mesh, was dried to less than 2% moisture and screened to -100 mesh.

The process development was carried out for the most part using tailings refloat products from Brunswick Mining and Smelting. Concentrates with higher lead and copper levels were prepared by blending lead concentrates from Brunswick Mining and Smelting and copper concentrates from Heath Steele Mines Ltd.

TABLE A1-1

RANGES OF COMPOSITION OF SAMPLES TESTED IN SULPHATION ROASTING (wt %)

	(1)	(2)	(3)	(4)	(5)
	Tailings Refloat Conc.	Bulk Conc.	High Cu Bulk Conc.	Zinc Conc.	Tailings Refloat and Ferrite Res.
Zn	22-32	27-31	30.5	51.2	32
Cu	0.4-0.9	0.7-0.9	4.5	0.5	0.4
Pb	2-4	10-14	4.8	2.0	2
Fe	22-26	18-20	21.5	9.9	26
S	36-40	34-35			
Co	0.015-0.025	0.020-0.023	0.053	0.006	0.002
Cd	0.060-0.065	0.053-0.065	0.060	0.097	0.055
As	0.3-0.5	0.3-0.5	0.27	0.75	0.4

(1) Series B2C, BMS, BMD (Tailings refloat from Brunswick Mining and Smelting)
(2) " BHL (Bulk concentrate, low in copper)
(3) " BHB, BHC, BHD (Bulk concentrate, high in copper)
(4) " BZC (Low-grade zinc concentrate)
(5) " BF (Tailings refloat and ferrite residue)

TABLE A1-2

CHEMICAL ANALYSES OF FEEDS TESTED IN SULPHATION ROASTING

CONCENTRATES	RUN #	Zn	Cu	Pb	Fe	S	Co (ppm)	Cd (ppm)	As	Ag (ppm)	Sn	Sb (ppm)	In (ppm)	Ni (ppm)	CaO	Al_2O_3	SiO_2	MgO	Ge (ppm)	Ga (ppm)	Cl	Hg
Zn tailings refloat II-2	BZC 1-17	22.80	0.22	1.67	28.50	42.76	-	-	-	-	-	-	-	-	0.30	0.67		0.03	-	-	-	-
" " " I	" 23-29	22.30	0.25	2.70	28.60	41.25	-	-	-	-	-	-	-	-	0.14	1.76		0.03	-	-	-	-
" " " II-1	" 33-40	22.70	0.22	2.46	28.50	42.70	-	-	-	-	-	-	-	-	0.28	0.94		0.08	-	-	-	-
" " " III	BHS 1-16	16.40	0.37	2.70	31.40	42.90	-	-	-	114	-	-	-	-	0.31	0.15	1.46	0.10	-	-	-	-
" " " V	BHD 1-22	29.50	0.98	3.23	23.42	40.05	-	-	-	136	-	-	-	-	0.28	0.21	0.38	0.10	-	-	-	-
" " " VI	" 23-36	29.00	1.16	3.38	23.70	39.50	-	-	-	-	-	-	-	-	0.36	0.19	0.62	0.11	-	-	-	-
" " " VII	" 37-45	29.50	1.28	3.70	22.50	39.10	230	650	0.44	94	-	-	-	-	0.35	0.21	0.86	0.11	-	-	-	-
" " " VIII (Blend 25% Zn + 51% Zn)	" 46-51	30.50	0.54	2.60	24.38	33.60	205	615	0.70	-	-	-	-	-	-	-	-	-	-	-	<0.01	trace
" " " IX (Blend 24% Zn + 51% Zn)	" 52	30.50	0.44	2.65	23.75	37.59	170	650	0.35	91	-	-	-	-	0.18	-	0.90	0.10	<3	8	-	-
" " " VII	" 53	29.50	1.28	3.70	22.50	39.10	230	650	0.44	94	-	-	-	-	0.35	0.23	0.86	0.11	-	-	-	-
" " " IX (Blend 24% Zn + 51% Zn)	" 54-61	30.50	0.48	2.65	23.75	37.59	170	650	0.35	91	-	-	-	-	0.24	-	0.90	0.10	3	8	-	-
" " " X (Blend 22% Zn + 51% Zn)	" 62-66	32.47	0.46	2.30	22.33	37.87	225	645	0.45	115	<0.01	<25	290	<100	0.16	-	0.68	0.10	3	16	-	-
Blend Zn tailings refloat X + Ferrite residue	BF 1-2	31.79	0.33	1.96	25.97	32.15	200	550	0.38	93	<0.01	<21	267	<90	0.15	-	0.38	0.09	-	-	-	-
Zn conc. (low grade)	BZC 1	51.20	0.50	2.00	15.90*	27.63*	60	970	0.20	75	<0.01	100	-	-	-	-	-	0.12	-	-	-	-
Blend Zn tailings refloat VII + Zn conc. + Pb conc.	BDL 1-4	30.80	0.74	10.64	18.40	34.39	200	625	0.33	240	<0.01	-	-	-	0.22	-	-	-	-	-	<0.01	trace
" " conc. + Pb conc.	" 5	26.75	0.92	14.30	19.40	34.14	230	530	0.45	240	<0.01	-	-	-	-	-	-	-	-	-	-	-
Blend Zn conc. + Cu-Pb conc.	BHS 1-2	28.75	5.12	9.05	19.38	33.00	500	560	0.25	250	<0.01	-	-	-	-	-	-	-	-	-	-	-
" " tailings refloat VII + Zn conc. + Cu-Pb conc.	BHC 1	30.50	6.80	3.00	19.00	32.56	520	580	0.26	260	<0.01	-	-	-	-	-	-	-	-	-	-	-
" " " " " " " " "	BHD 1	30.50	4.48	4.00	21.50	35.15	530	600	0.30	270*	<0.01	-	-	-	0.69*	0.20*	0.49*	0.34*	-	-	-	-

(*) Calculated Values

122 Process Development

In the early stages in the development of the Process, consideration was given to the use of a slurry feeding system for the reactor. The reasoning being that spent zinc electrolyte could be used as the liquid medium, and reactor cooling agent, and hence the quantity of electrolyte required to be neutralized would be reduced. This was the approach followed earlier by the St. Joseph Lead Company in their attempt to develop a sulphation-roast process for zinc (1) as well as by the Dowa Co. in Japan in their commercial zinc-copper sulphation plant (2). The principal problems encountered by Dowa and in the St. Joseph Lead program, i.e. high iron levels in leach solutions going to purification and poor zinc recoveries, were found to be principally due to an enhancement of ferrite formation which was identified as a problem inherent to slurry feeding. As a result, the slurry feed approach was eventually abandoned in favour of dry feeding.

The main responses of the roasting operation were the ferrite formation, measured by x-ray diffraction, and the zinc extraction in both water and dilute acid, monitored by means of small, 50 g calcine sample control leaches in water (16% solid, 25°C, 1 hr) and in dilute acid, (16% solid, 25 gpl H_2SO_4, 25°C, 1 hr). Under these conditions the control leaches produce solutions with 30 to 35 gpl Zn.

The following summarizes the experimental programs for both feed systems.

TABLE A1-12
HIGH COPPER BULK CONCENTRATES CHEMICAL COMPOSITION

Feed	Wt % Zn	Cu	Pb	Fe
BHB	28.75	5.12	9.05	19.38
BHC	30.50	6.80	3.00	19.00
BHD	30.50	4.48	4.00	21.50

Roasting conditions used in all three tests were the same as those used for the tailings refloat products and also for the bulk concentrates, as follows:

temperature = 685°C
air factor = 1.85
feed rate = 5.0 (kg/hr)
retention time = 6.6 (hrs)

Defluidization problems were experienced with run BHB with 5.12 wt% copper and 9.05 wt% lead. Although the bed did not sinter or harden, the 'stickiness' was sufficient to avoid proper fluidization with an air velocity of 19.2 cm/sec. A similar, although reduced effect, was experienced with run BHC with copper at 6.80 wt% and lead at 3.00 wt%. With the copper level lowered to 4.5 wt% and lead at 4.8 wt%, as in run BHD, the stickiness problem disappeared. This particular feed was run continuously, on a two shift basis, for two days without difficulty. The calcine obtained was low in zinc oxysulphate and ferrite, 3.1 wt% and 8.9 wt% respectively, which is consistent with the observations from the tailings refloat tests, i.e., that high copper content could reduce the level of zinc oxysulphate in calcines and as a consequence limit the formation of ferrite.

The stickiness problem associated with 'relatively' high copper with lead is most probably a result of a low melting eutectic involving both copper and lead and this may not be a problem in commercial reactors where air velocities are greater. However, information on the lead-copper-sulphate system could not be found in the literature and hence more test work should be undertaken to establish the working range for copper and lead in the sulphation process.

Results of interlocked tests with BMD material using HAL solution equivalent to that which would be obtained from a neutral leach residue from a calcine containing 15 wt% ferrite and 1.5 wt% sphalerite are given in Tables A1-9, A1-10 and A1-13.

TABLE A1-13
RESULTS OF ROASTING TESTS WITH HIGH COPPER BULK CONCENTRATE

Run	Metal Extraction %				
	dil. acid leach(1)		overall(2)		
	Zn	Cu	Zn	Cu	Fe(gpl)
BHB-1	--------- rejected due to defluidization of bed --------				
BHC-1	--------- rejected due to defluidization of bed --------				
BHD-1 & 2	87.39	86.28	98.99	98.90	0.40

(1) Primary extraction from feed, leach at 25 gpl H_2SO_4, 1 hour, 25°C, 16% solids.
(2) Calculated assuming 92% extraction Zn and Cu in HAL.

.7 Effect of Increased Zinc

A zinc concentrate containing 51 wt% zinc was subjected to one preliminary test to determine the effect of increased zinc. The results, given in Tables A1-9 and A1-10 and summarized in Table A1-14 were rather poor, with less than 66% primary extraction of zinc in the dilute acid leach. This is an artificially low result due to insufficient acid in the control leach for efficient oxysulphate leaching. The oxysulphate level was high as was the quantity of ferrite at 44.0 wt% and 29.0 wt% respectively, possibly due to the low SO_2 partial pressure in the bed resulting from the low pyrite content of the feed. More test work would obviously be required to re-establish optimum roasting conditions for zinc-rich concentrates.

TABLE A1-14
RESULTS OF ZINC CONCENTRATE ROASTING TEST

Run	Feed			Metal Extraction %				
				water(1)		acid(2)		
	%Zn	%Cu	%Pb	Zn	Cu	Zn	Cu	Fe(gpl)
BZC-1	51.2	0.5	2.0	39.11	0.61	65.96	1.17	0.00

(1) Primary extraction in water. Leach: 1 hr, 25°C, 16% solids.
(2) Primary extraction in acid. Leach: 25 gpl H_2SO_4, 25°C, 1 hr, 16% solid.

.8 Ferrite Resulphation Test

Two roasting tests were carried out to gain information on the resulphation of ferrite. In the one case, RF-1, a mixture of 30% zinc tailings refloat product and -100 mesh synthetic ferrite was prepared to give a blend containing 15 wt% ferrite. Synthetic HAL solution (approximating the composition which would be derived from a calcine containing

15 wt% ferrite and 1.5 wt% sphalerite) was used as the coolant. A second test, BF-2, was carried out using the same proportion of ferrite to tailings refloat product, but with the feed introduced to the roaster as a slurry with the HAL solution.

The leaching results indicate that, without the use of promoters, resulphation of ferrite is only partially achieved, i.e. approximately 47.8% resulphation with the dry feed, and 66.7% with the slurry feed (Tables A1-9, A1-10 and A1-15). The higher resulphation of ferrite in the slurry feed system is probably a result of prior dissolution of ferrite in the HAL solution. Higher sulphation of ferrite values using ferrite residues and pyrite concentrate blends, plus Na_2SO_4 promoters were reportedly achieved by Det Norske Zinkkompani in Norway (8) and by the Akita Zinc Co. in Japan (9) both in commercial operations. This resulphation process has apparently been abandoned at both plants.

TABLE A1-15
RESULTS OF FERRITE RESULPHATION TESTS

Run	Feeds (%)			Metal Extractions %		
	Tailings Refloat		Ferrite	From Ferrite Res.	From Tailings Refl.(3)	
	Zn	Cu	Zn	Zn	Zn	Cu
BF-1	36.62	0.46	27.11(1)	47.84	85.60	79.54
BF-2	32.62	0.46	27.11(2)	67.68	85.61	63.73

(1) Blend of dry -100 mesh tailings refloat (85 wt%) + ferrite (15 wt%).

(2) Tailings refloat dry -100 mesh (85 wt%) + ferrite slurried with HAL solution (15 wt%).

(3) Primary metal extraction in acid. Leach: 25 gpl H_2SO_4, 1 hr, 25°C, 16% solid.

123 Considerations for Plant Scale-up

Based on the 14-inch diameter reactor system, the following roasting conditions were chosen for scale-up to a commercial unit:

roasting temperature = 685°C
retention time of solids = 5.5 (hrs)
air factor = 1.8
feed rate = 4.1 (MT/m^2-day)

.1 Roasting Temperature

The roasting temperature was established at 685°C to limit the amount of soluble iron in the neutral leach. However, since at least part of the iron solubilized appears to be due to incomplete decomposition of the HAL solution in shortcircuited pellets, it is possible that in a commercial unit, where shortcircuiting would be less of a problem, the roasting temperature could be reduced, perhaps to 675°C or lower. The lower roasting temperature would also help to decrease the amount of ferrite in the calcine.

.2 Retention Time

Retention time is the limiting factor determining the amount of unreacted sphalerite in the calcine. However, as retention time is to some extent a function of the size of the reactor, it is possible that less than the specified 5.5 hours retention will be possible in commercial reactors without increasing the amount of unreacted sphalerite in the calcine above 1.5 wt%. Lower retention time means increased reactor capacity per unit area and hence lower capital costs per unit of recovered zinc.

.3 Air Factor

Again, the air ratio to some extent is a function of reactor size, i.e. the smaller the reactor the greater the required air velocity to maintain proper bed fluidization. This, plus consideration of existing commercial sulphation roasters, would indicate that the proposed zinc sulphation roaster might operate at an air factor of 1.8 or lower.

The air factor is also an important variable in determining the (SO_2+SO_3) strength in the roaster off-gas and the dust load. Also, as heat is required to preheat the air inside the reactor, any reduction in the air factor will effectively increase the capacity of the roaster for consuming either HAL solution or spent electrolyte.

An air ratio of 1.8 was selected for scale-up purposes. At a bed depth of 1.5 m at 685°C this corresponds to a gas velocity of 75 cm/sec compared with 21 cm/sec for the 14-inch pilot reactor under the same roasting conditions.

.4 Feed Rate

For a given reactor diameter, the feed rate is determined by the fluidized bed height. Although bed depths can extend up to 3 m, a bed height of 1.5 m was chosen for calculation purposes. This corresponds to the bed levels commonly employed in conventional dead roasting of zinc concentrates.

.5 Thermal Balance

The consumption of all HAL solution and wash water from the ferrite residue leach in the roaster is an essential part of the RPC process. Thermal balances performed on all zinc products tested show that for 15 wt% ferrite in the calcines, the heat excess generated in the bed alone, not considering the sensible heat of the hot gases inside the reactor, is sufficient to consume all HAL solution and wash water (Appendix A3). For example, for the tailings refloat product the capacity of the reactor for decomposing HAL solution is 0.814 ton of HAL solution per ton of sulphide feed. The basis of this solution is 100 gpl Fe and 100 gpl Zn (Figure A1-31), with a specific gravity of 1.53 g/cm^3, containing 0.53 tons H_2O per ton feed. This represents a 15 % excess capacity in the reactor over that required to consume all HAL solution from a calcine containing 15 wt% ferrite and 1.5 wt% sphalerite.

The above thermal calculations are in agreement with published data for the Chambishi copper-cobalt sulphation plant (5) and with the experimental results achieved in the St. Joseph Lead pilot trials with zinc concentrates (1). At Chambishi, spent electrolyte is consumed in the reactor at the rate of 1.07 tons of water per ton of feed, that is accomplished with a temperature drop in the freeboard from 680°C to 530°C. At the Hecla copper sulphation plant in Arizona (6), gases exit from the reactor at 650°C. The slurry gun is only 0.95 m from the fluidized bed surface and there is virtually no cooling of the gases in the freeboard of the reactor. (Roasting is done at 680°C.) In this plant, the water vaporized to feed ratio is 0.61.

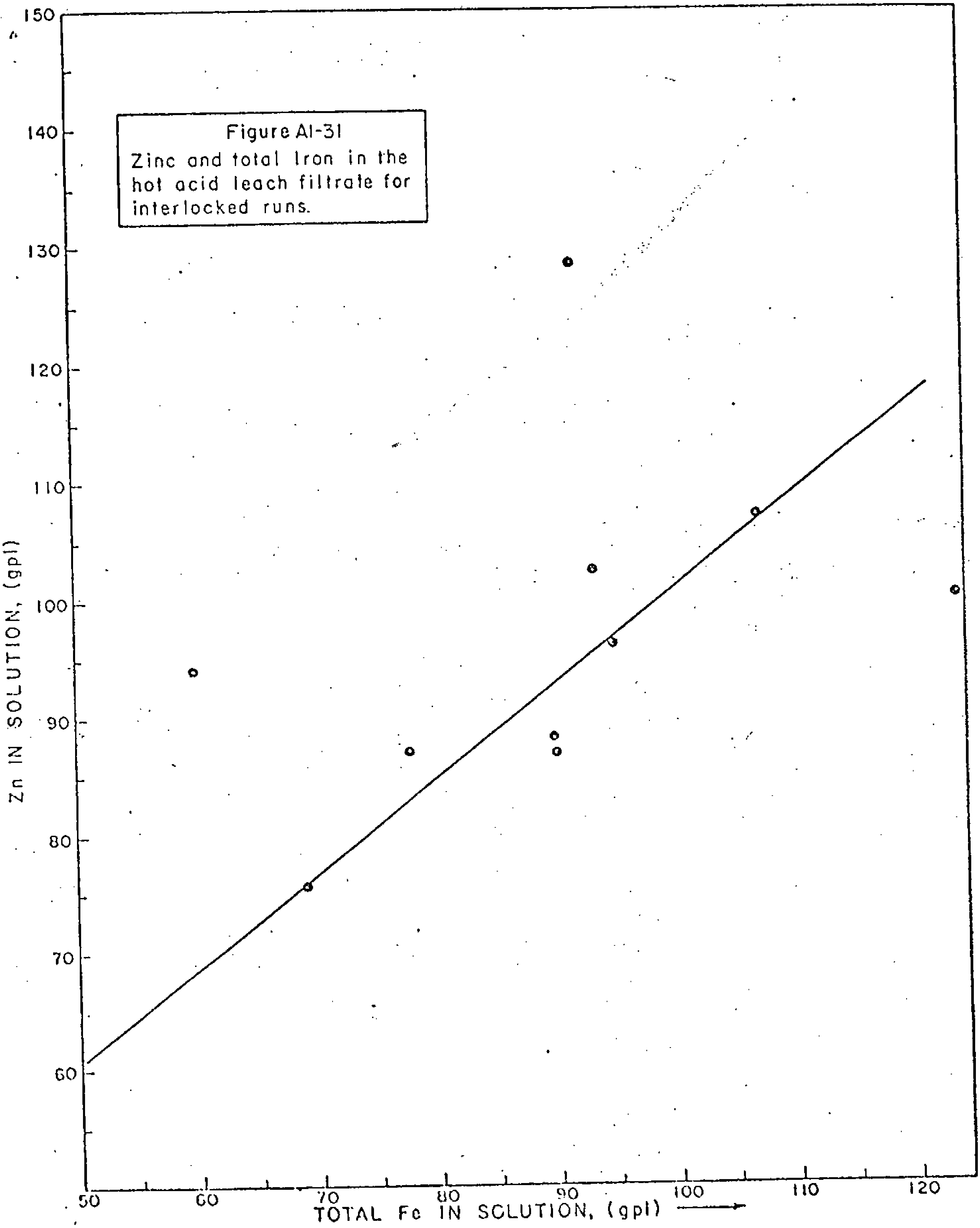


Figure AI-31
Zinc and total Iron in the hot acid leach filtrate for interlocked runs.
150
140
130
120
110
100
90
80
70
60
Zn IN SOLUTION, (gpl)
50
60
70
80
90
100
110
120
TOTAL Fe IN SOLUTION, (gpl)

Sulphation and oxidation reactions for copper concentrates (chalcopyrite) generate approximately 85% of the heat evolved from the roasting of tailings refloat products and 90% of the heat generated for the bulk concentrate on a per ton of feed basis. In the St. Joe Lead work spent electrolyte was consumed in the reactor at the equivalent rate of 0.8 to greater than 2 tons of water per ton of feed, depending on the feed and operating conditions employed.

Additional heat capacity in the roaster would also be derived with a decrease in the air factor. A decrease in Q from 1.8 to 1.6 would increase the capacity of the reactor to consume HAL solution by approximately 20%. The relationship of air factor, freeboard gas temperature, and HAL decomposition capacity in commercial reactors is shown in Figure A1-32. The calculations are based on a tailings refloat product, which, when roasted, yields a calcine containing 15 wt% ferrite and 1.5 wt% sphalerite.

It can be seen from Figure A1-32 that the reactor's capacity to consume the HAL solution increases rapidly with the decrease of the freeboard gas temperature. For example, at Q = 1.6, and allowing the off-gases to cool down from 685°C to 500°C, the capacity of the roaster to decompose the HAL solution increases by 83%, this represents an increase in thermal capacity of 28 kg HAL solution/°C-ton feed. However, excess consumption of heat in the freeboard to consume HAL solution will decrease the temperature of the exit off-gas and thereby reduce the production of steam in the waste heat boilers as shown in Figure A1-32.

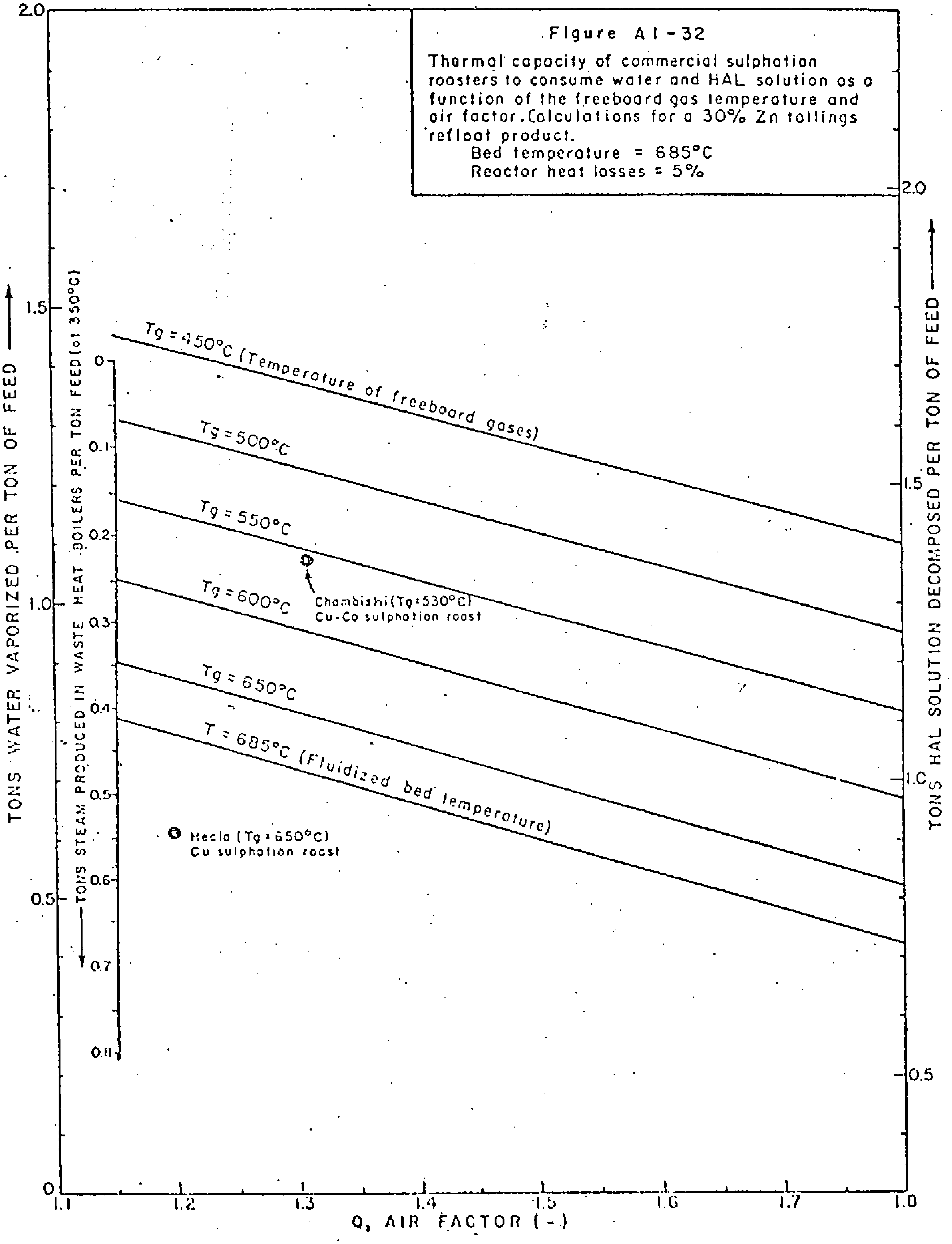


Figure A1-32

Thermal capacity of commercial sulphation roasters to consume water and HAL solution as a function of the freeboard gas temperature and air factor. Calculations for a 30% Zn tailings refloat product.
Bed temperature = 685°C
Reactor heat losses = 5%

The roasting of the bulk concentrate generates about 7% less heat due to the lower levels of pyrite in the feed and higher proportion of galena present.

The thermal losses are estimated as 5% of the reaction heat, a figure usually achieved in commercial units that recover the heat generated in the reactor. In the small 14-inch pilot unit, measured thermal losses were approximately 13-17%. The increase in heat losses from the roaster rapidly decreases the excess heat available (from the bed), for example, for a 15% heat loss, the capacity for consuming the HAL solution decreases by about 38%. During the interlocked runs, external heat was added to compensate for the difference in heat losses from an equivalent commercial unit and achieve the same ratio between feed and HAL solution consumed.

.6 Calculation of Reactor Diameter

The reactor diameter for a roaster is calculated from the following relationship:

$$Dr = 2\left[\left(\frac{1}{\pi}\right)\left(\frac{1}{1-\sigma}\right)\left\{\frac{\bar{t}(F_o\gamma+R)}{L_f\ \rho_c}\right\}\right]^{0.5}$$

The variables are defined as follows:

Dr = reactor diameter (m)
F_o = feed rate, (dry basis) (variable) (TPH)
R = dissolved solids in HAL solution = $0.123F_o$ (TPH)
$\bar{t}$ = average retention time of solids = 5.5 (hrs)
L_f = fluidized bed depth = 1.5 (m)
ρ_c = average bulk density of calcines = 1.15 (T/m^3)
σ = gas fraction in fluid bed = 0.35 (-)
γ = weight transformation coefficient = 1.095 (-)

The gas fraction in the fluidized bed (σ) was calculated using a theoretical model while the experimental values for σ determined in the pilot reactor are given in Figure A6-2.

Values for roaster diameter (Dr) as a function of roaster capacity for retention times ($\bar{t}$) of 5.5 and 6 hours are shown in Figure A1-33. The calculated capacity is approximately 80% greater than for the Dowa (2) zinc-copper sulphation roaster which operated commercially for a short period during the late 1950's, and 30% smaller than the Hecla copper sulphation plant.

Since the scale-up calculation is based on conservative estimates for $\bar{t}$, L_f and Q, it is possible that for a given reactor diameter, the calculated capacity for a commercial plant may be increased by as much as 25-30%.

The capacity of a fluidized bed sulphation reactor, in addition to relating to the variables outlined above, also appears to be largely a function of independent design parameters. For example, comparison of reactors designed by two prominent manufacturers, Dorr-Oliver and Lurgi, show a large difference in capacity per unit area of bed as shown in Figure A1-34. Copper-cobalt sulphation roasters designed by Dorr-Oliver, i.e. Rhokana, U. Miniere, Ndola 2, Ndola 1 and Hoboken all have capacities of less than 4 MT/m^2-day, whereas similar plants built by Lurgi, i.e. Chambishi, and Hecla have capacities greater than 6 MT/m^2-day. Similarly the roaster capacity (2.6 MT/m^2-day) for

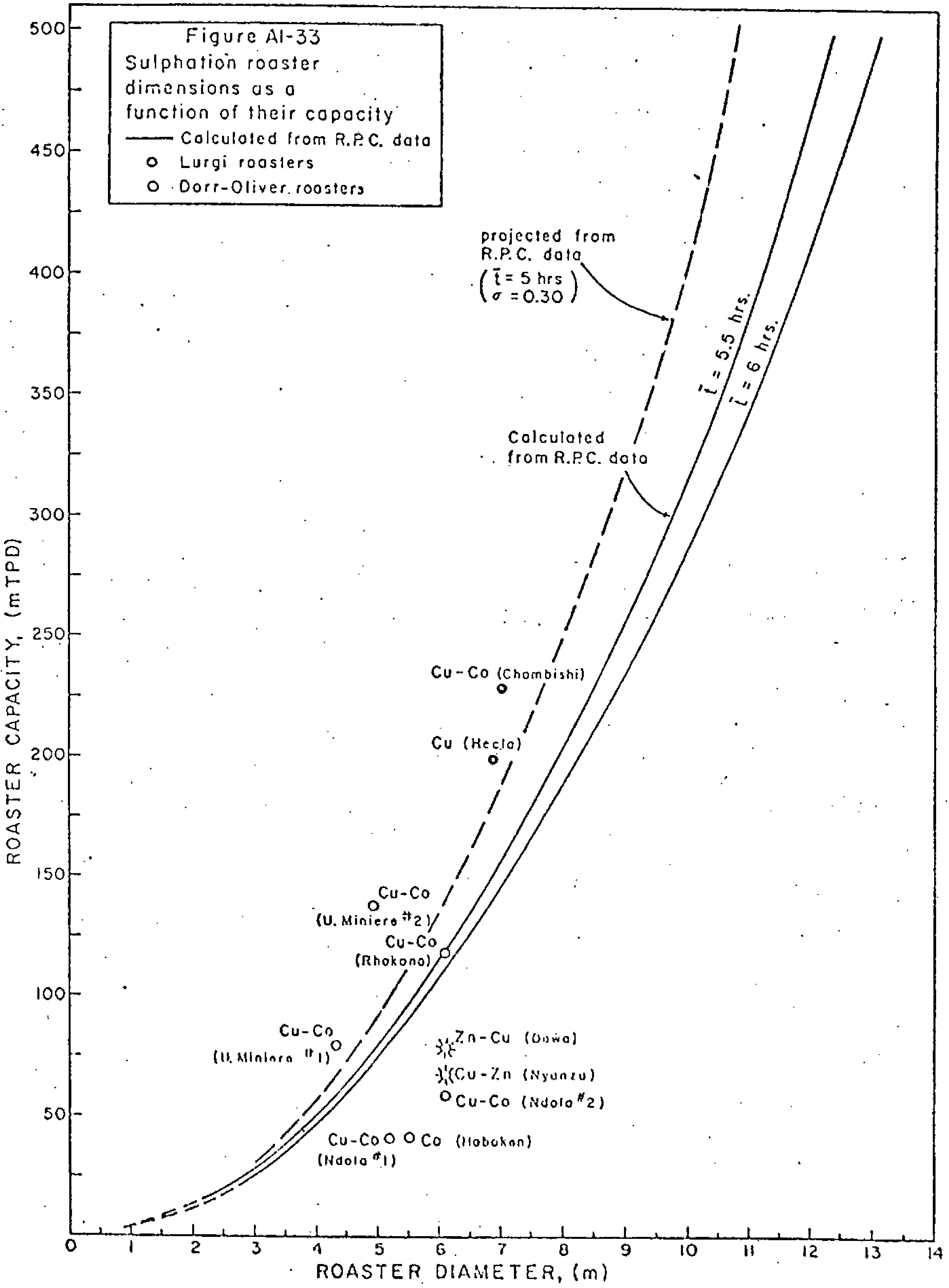


Figure A1-33
Sulphation roaster dimensions as a function of their capacity

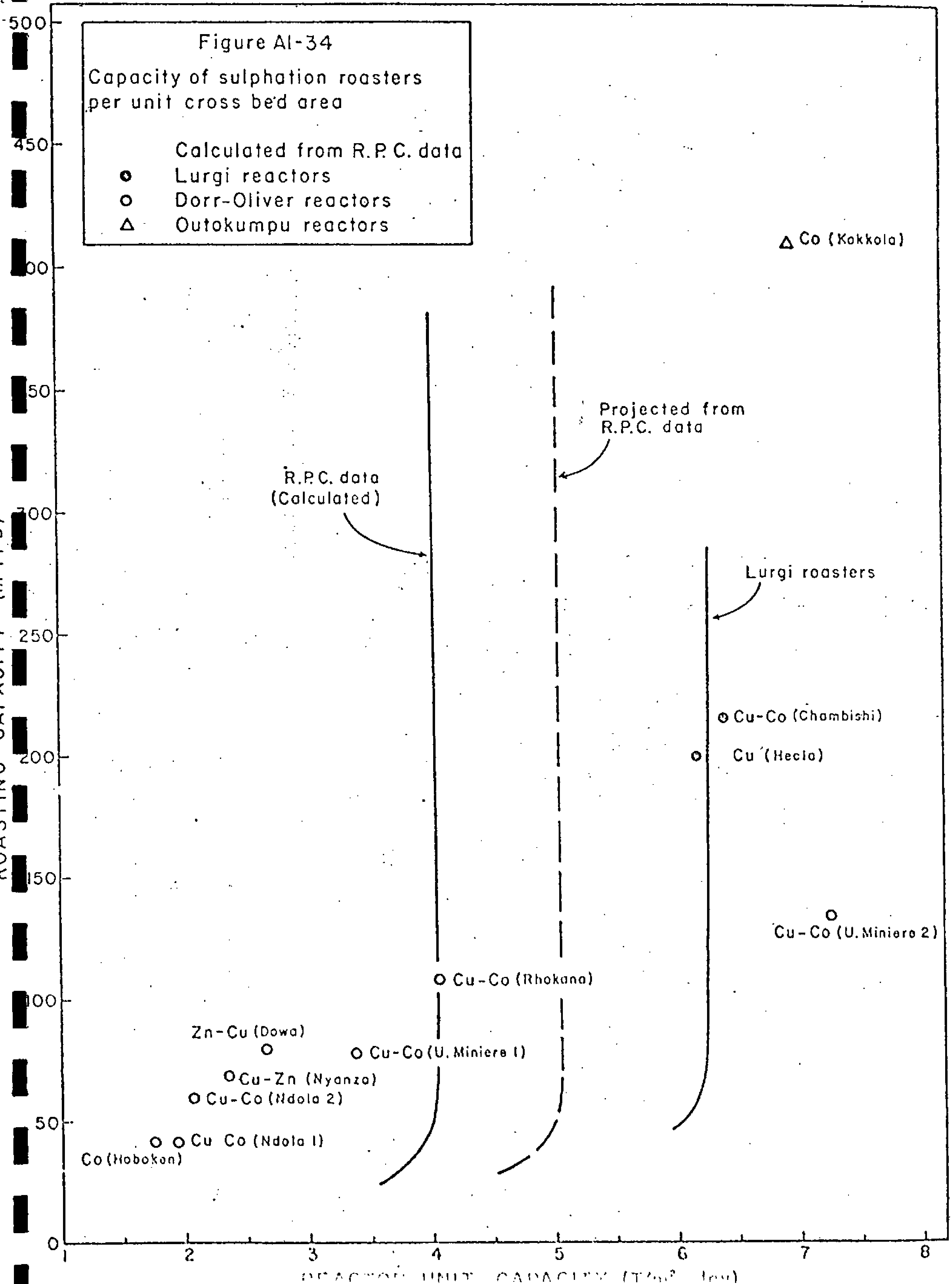


Figure Al-34

Capacity of sulphation roasters per unit cross bed area the Dowa Zn-Cu plant, which used Dorr-Oliver roasters, is small in comparison to what would be required for a sulphation roast zinc plant as determined using the present experimental data (4.1 MT/m^2-day).

.7 Elutriated Solids Collection and Gas Cleaning

The elutriated solids collection equipment used in a commercial sulphation roaster would be similar to that used in conventional dead roast systems.

A one or two stage hot cyclone system with a combined collection efficiency of 90% would be employed with discharge of elutriated material back to the bed through rotary star valves. The off-gases exiting the cyclones at 650°C or less (depending on the heat recovered in the freeboard for decomposition of the HAL solution) are further cooled to 350°C in a waste heat boiler. Since the entrance temperature of the waste heat boilers is low (relative to a dead roast system), no radiation section is required in the waste heat boiler. The waste heat boiler with no radiation zone would be similar to that presently employed by Outokumpu in their cobalt sulphation plant (7). The waste heat boiler would recover from 0.4 to 0.6 tons of steam per ton of feed; depending upon the composition of the feed and the gas temperature. This satisfies a sizable amount of the total steam requirements in the hydrometallurgy section (Appendix A4). The waste heat boiler would also collect about 2% of the total dust elutriated from the reactor.

An electrostatic precipitator operating at 350 to 300°C, and similar to that employed at the cobalt sulphation plant of Outokumpu, is the final gas cleaning stage. Approximately 8% of the elutriated dust is collected in the electrostatic precipitator. This material and that derived from the cyclones and waste heat boilers represents an overall collection efficiency of 99.5%. Dust collected from the cyclones, waste heat boilers and electrostatic precipitators is relatively high in soluble iron and hence, in all probability, must be recycled to the roaster.

Operation of the electrostatic precipitator at 350°C versus the 400 to 450°C employed in the mini-pilot runs may result in a buildup in arsenic in the calcine due to the recycle of precipitator dust to the roaster. It is proposed that, if further testing indicates such a buildup, the electrostatic precipitator dust be "neutral" leached separately to avoid dissolution of the As_2O_3 and flocculated and thickened. The residue would be incorporated with the scrubber acid, and the clarified liquor recycled to the roaster with the HAL solution or used as a base solution for the hot acid leach step.

Off-gases exiting from the electrostatic precipitator are cooled in a scrubber by vaporizing water and condensing the resulting acid water before entering the acid plant. Virtually all impurities, such as arsenic, selenium, chlorine and mercury, which are volatilized during roasting are removed in the acid water bleed.

APPENDIX A3

OVERALL THERMAL BALANCE FOR THE FLUIDIZED BED SYSTEM-TAILINGS REFLOAT PRODUCT

APPENDIX A3

OVERALL THERMAL BALANCE FOR THE FLUIDIZED BED SYSTEM-TAILINGS REFLOAT PRODUCT

A3.1 OPERATIONS CONDITIONS

Feed rate = 37.50 TPH (dry base)
Temperature = 685°C
Air factor = 1.80

A3.2 FEED AND CALCINES COMPOSITION

321 Feed Composition

900.23 TPD (dry base)
(Zn,Fe)S = 456.23 TPD = 4.7421×10^6 (moles/day)
$CuFeS_2$ = 15.60 " = 8.35×10^4 "
PbS = 37.43 " = 1.561×10^5 "
FeS_2 = 352.16 " = 2.9350×10^6 "

322 Calcine Composition

1191.63 TPD:
$ZnSO_4$ = 565.09 TPD = 3.5010×10^6 (moles/day)
$ZnO.2ZnSO_4$ = 106.06 " = 2.624×10^5 "
$ZnO.Fe_2O_3$ = 129.50 " = 5.372×10^5 "
(Zn,Fe)S = 10.63 " = 1.105×10^5 "
$PbSO_4$ = 47.20 " = 1.559×10^5 "
$CuSO_4$ = 15.36 " = 0.962×10^5 "
Fe_2O_3 = 274.60 " = 1.7195×10^6 "

A3.3 AIR REQUIREMENT *

Oxygen required to form desired composition according to:

$$(0.8712\ ZnS + 0.1288\ FeS) + 1.968 O_2 \rightarrow 0.8712\ ZnSO_4 + 0.0644\ Fe_2O_3 + 0.1288\ SO_2$$

$$CuFeS_2 + 3.75 O_2 \rightarrow CuSO_4 + 0.5\ Fe_2O_3 + SO_2$$

[illegible] has a Zn:Fe ratio

$PbS + 2O_2 \rightarrow PbSO_4$

$FeS_2 + 2.75O_2 \rightarrow 0.5\ Fe_2O_3 + 2SO_2$

$$\begin{aligned} \dot{n}O_2 &= 0.0173\ (3.76/183.51) + 0.5068\ (1.968/96.207) \\ &\quad + 0.0416\ (2.0/239.25) + 0.3912\ (2.75/119.97) \\ &= 2.003 \times 10^{-2}\ (\text{moles } O_2/\text{gr concentrate}) \end{aligned}$$

$$q = 2.130 \times 10^{3}\ (m^3 \text{ air/Ton conc.})\ (\text{st. cond.})$$

For an air factor of Q = 1.8 (80% excess air into the fluidized bed),

$$q = 3.834 \times 10^{3}\ (m^3 \text{ air/Ton conc.})$$

or

$$\begin{aligned} Q &= 3.834 \times 10^{3} \times 9.0023 \times 10^{2} \\ &= 3.451 \times 10^{6}\ (m^3/\text{day})\ (\text{st. cond.}) \end{aligned}$$

A3.4 HEAT GENERATED AND CONSUMED

341 Heat Generated by Reactions

The values of Table A3-1 have been used to calculate the heat of reactions at 685°C (958°K).

TABLE A3-1

STANDARD HEATS OF FORMATION AND HEAT CAPACITIES

Compound	$\Delta H^\circ_{298^\circ K}$ (K cal/mole)	C_p (cal/mole-°K)
ZnS	- 48.2	$12.16 + 1.24 \times 10^{-3}\ T$
$ZnSO_4$	-234.9	$21.90 + 18.2 \times 10^{-3}\ T$
$ZnO.2ZnSO_4$	-551.3	75.5
$ZnO.Fe_2O_3$	-279.5	$34.3 + 13.3 \times 10^{-3}\ T$
FeS	- 22.8	$12.20 + 2.38 \times 10^{-3}\ T$
FeS_2	- 42.4	$17.88 + 1.32 \times 10^{-3}\ T$
Fe_2O_3	-196.3	$23.49 + 16.6 \times 10^{-3}\ T$
$CuFeS_2$	- 76.2	$22.89 + 40.33 \times 10^{-3}\ T$

TABLE A3-1 (Cont'd)

Compound	$\Delta H^\circ_{298^\circ K}$ (K cal/mole)	C_p (cal/mole-°K)
$CuSO_4$	-184.0	$18.77 + 17.20 \times 10^{-3}\ T$
PbS	- 22.5	$10.66 + 3.92 \times 10^{-3}\ T$
$PbSO_4$	-219.5	$10.96 + 31.0 \times 10^{-3}\ T$
SO_2 (g)	- 71.0	$10.38 + 2.54 \times 10^{-3}\ T$
O_2	-	$7.16 + 1.0 \times 10^{-3}\ T$
H_2O (g)	- 68.3	$7.30 + 2.46 \times 10^{-3}\ T$

.1 Sphalerite

To form $ZnSO_4$:

$$(0.8712\ ZnS + 0.1288\ FeS) + 1.968\ O_2 \rightarrow$$
$$0.8712\ ZnSO_4 + 0.0644\ Fe_2O_3 + 0.1288\ SO_2 \text{ ---------- } \quad (1\text{-a})$$

Heat of reaction at 958°K calculated according:

$$\Delta H^\circ_{298^\circ K} = -(0.8712 \times 234.9 + 0.0644 \times 196.3 + 0.1288 \times 71) + (0.8712 \times 48.2 + 0.1288 \times 22.8)$$

$$\Delta H^\circ_{298^\circ K} = -180.63 \text{ (K cal/mole (Zn,Fe)S)}$$

(assuming no heat of solution between ZnS and FeS in the marmatitic sphalerite)

$$\Delta C_p = (19.08 + 15.86 \times 10^{-3}\ T) + (1.51 + 1.07 \times 10^{-3}\ T) + (1.34 + 0.33 \times 10^{-3}\ T) - (10.59 + 1.08 \times 10^{-3}\ T) - (1.57 + 0.31 \times 10^{-3}\ T) - (14.09 + 1.97 \times 10^{-3}\ T)$$

$$\Delta C_p = -4.32 + 13.9 \times 10^{-3}\ T \text{ (cal/mole (Zn,Fe)S)}$$

$$\Delta H^\circ_T = \int(-4.32 + 13.9 \times 10^{-3}\ T)\,dT$$
$$= -4.32T + 6.95 \times 10^{-3}\ T^2 + \Delta H_o$$

$$\Delta H_o = -180{,}630 + 4.32 \times 298 - 6.95 \times 10^{-3} \times \overline{298}^2$$
$$= -179{,}960 \text{ cal/mole (Zn,Fe)S}$$

$\Delta H°_T = -179{,}960 - 4.32T + 6.95 \times 10^{-3}\ T^2$

Therefore, at 958°K:

$\Delta H°_{958°K} = -177.72$ (K cal/mole (Zn,Fe)S)

or

$\Delta H°_{958°K} = -203.99$ (Kcal/mole $ZnSO_4$)

To form $ZnO.ZnSO_4$:
$(0.8712\ ZnS + 0.1288\ FeS) + 1.532\ O_2 \rightarrow$
$0.2904\ (ZnO.2ZnSO_4) + 0.0644\ Fe_2O_3 + 0.1288\ SO_2$ ---------- (1-b)

$\Delta H°_{298°K} = -136.96$ (K cal/mole (zn,Fe)S)
$\Delta C_p = 1.66 - 1.52 \times 10^{-3}\ T$ (cal/mole °K)
$\Delta H_o = -137{,}352$ (cal)

$\Delta H°_T = -137{,}352 + 1.66T - 7.62 \times 10^{-3}\ T^2$ (cal/mole (Zn,FeS))

$\Delta H°_{958°K} = -136.46$ (K cal/mole (Zn,Fe)S)

or

$\Delta H°_{958°K} = -469.90$ (K cal/mole $ZnO.2ZnSO_4$)

To form $ZnO.Fe_2O_3$:
$ZnO.2ZnSO_4 + Fe_2O_3 \rightarrow ZnO.Fe_2O_3 + 2ZnSO_4$ ------------------ (1-c)

$\Delta H°_{298°K} = -1.71$ (K cal/mole $ZnO.2ZnSO_4$)
$\Delta C_p = -20.9 + 33.1 \times 10^{-3}\ T$ (cal/mole-°K)
$\Delta H_o = +3048$ (cal)

$\Delta H°_T = +3048 - 20.9T + 16.55 \times 10^{-3}\ T^2$ (cal/mole $ZnO.2ZnSO_4$)

.2 Chalcopyrite

$CuFeS_2 + 3.75\ O_2 \rightarrow CuSO_4 + 0.5\ Fe_2O_3 + SO_2$ ---------- (2)

$\Delta H°_{960°K} = -309.21$ (K cal/mole $CuFeS_2$ or $CuSO_4$)
(Incra Tables)

.3 Galena

$PbS + 2O_2 \rightarrow PbSO_4$ ------------------------------ (3)

$\Delta H°_{298°K} = -197.0$ (K cal/mole PbS)
$\Delta C_p = -14.02 + 25.08 \times 10^{-3}\ T$ (cal/mole °K)
$\Delta H_o = -156{,}390$ (cal)

$\Delta H°_T = -156{,}390 - 14.02T + 12.5 \times 10^{-3}\ T^2$ (cal/mole PbS)

$\Delta H°_{958°K} = -158.35$ (K cal/mole PbS or $PbSO_4$)

.4 Pyrite

$FeS_2 + 2.75O_2 \rightarrow 0.5\ Fe_2O_3 + 2SO_2$ -------------------- (4)

$\Delta H°_{298°K} = -197.90$ (K cal/mole FeS_2)
$\Delta C_p = -5.06 + 9.31 \times 10^{-3}\ T$ (cal/mole FeS_2)
$\Delta H_o = -196{,}706$ (cal)

$\Delta H°_T = -196{,}706 - 5.06T + 4.66 \times 10^{-3}\ T^2$ (cal/mole FeS_2)

$\Delta H°_{958°K} = -197.29$ (K cal)

.5 Total Heat Generated

.51 From Sphalerite

Total $ZnSO_4$ in calcines = 3.5010 x 10^6 (moles/day)
Total $ZnSO_4$ produced by reaction (1-c)= 5.372 x 10^5 (moles/day)
Net $ZnSO_4$ produced by reaction (1-a)= 2.427 x 10^6 (moles/day)

$\Delta H_1 = -203.99 \times 2.427 \times 10^6 = -4.9508 \times 10^8$ (K cal/day)

According to reaction (1-b) to form the total $ZnO.2ZnSO_4$ present in the calcines plus the $ZnO.2ZnSO_4$ to form the $ZnO.Fe_2O_3$ present in the calcines:

$ZnO.2ZnSO_4$ in calcines = 2.6238 x 10^5 (moles/day)
$ZnO.2ZnSO_4$ to form $ZnO.Fe_2O_3$ = 5.3719 x 10^5 (moles/day)
Total $ZnO.2ZnSO_4$ formed = 7.9957 x 10^5 (moles/day)

$\Delta H_2 = -469.90 \times 7.9957 \times 10^5 = -3.7572 \times 10^8$ (K cal/day)

According to reaction (1-c) to form the total $ZnO.Fe_2O_3$ present in the calcines:

$\Delta H_3 = -1.79 \times 5.372 \times 10^5 = -9.62 \times 10^5$ (K cal/day)

.52 Chalcopyrite

According to raction (2), for the total $CuFeS_2$ present in the feed:

$\Delta H_4 = -309.21 \times 8.35 \times 10^4 = -2.582 \times 10^7$ (K cal/day)

.53 Galena

According to reaction (3), for the total PbS present in the feed:

$\Delta H_5 = -158.35 \times 1.56 \times 10^5 = -2.470 \times 10^7$ (K cal/day)

.54 Pyrite

According to reaction (4), for the total FeS_2 present in the feed:

$\Delta H_6 = -197.29 \times 2.935 \times 10^6 = -5.7905 \times 10^8$ (K cal/day)

(the total Fe_2O_3 present in the calcine is formed by oxidation of the FeS_2, $CuFeS_2$ and remnant FeS from the sphalerite).

.55 Total Heat Generated

$$\Delta H_R = \sum_{i=1}^{6} \Delta H_i = -1.5014 \times 10^9 \text{ (K cal/day)}$$

342 Heat Consumed in the System

.1 Heat Losses

The reactors are insulated to achieve 5% (as maximum) of heat losses (from the total heat generated by the reactions). Therefore,

$\Delta H_7 = +7.507 \times 10^7$ (K cal/day)

.2 Preheat the Fluidizing Air

$Q = 3.451 \times 10^6$ (m^3 air/day) (air blasted at 60°C after compression)

$= 3.234 \times 10^7$ (moles O_2/day)

$= 1.2171 \times 10^8$ (moles N_2/day)

Oxygen:

$H_T - H_{298°K} = -2313 + 7.16T + 0.50 \times 10^{-3} T^2$ at 958°K

$H_{958} - H_{333°K} = +4605$ (cal/mole O_2)

Total to heat O_2 = 1.489×10^8 (K cal/day)

Nitrogen:

$H_T - H_{298°K} = -2031 + 6.66T + 0.51 \times 10^{-3} T^2$ at 958°K

$H_{958} - H_{333°K} = +4395$ (cal/mole N_2)

Total to heat $N_2 = 5.345 \times 10^8$ (K cal/mole)

Total heat required to preheat the air:

$\Delta H_8 = +6.8346 \times 10^8$ (K cal/day)

.3 Preheat the Feed

Entalphies calculated according to:

ZnS:

$$H_{958} - H_{298°K} = \int_{298}^{958} (12.16T + 0.62 \times 10^{-3} T^2)\, dT$$

$= +8.54$ (K cal/mole)

Total heat $= +8.54 \times 4.1313 \times 10^6 = +3.527 \times 10^7$ (K cal/day)

FeS:

$H_{958} - H_{298°K} = +9.04$ (K cal/mole)

Total heat $= +9.04 \times 6.1078 \times 10^5 = +5.521 \times 10^6$ (K cal/day)

$CuFeS_2$:

$H_{958} - H_{298°K} = +33.1$ (K cal/mole)

Total heat $= +33.1 \times 8.35 \times 10^4 = 2.764 \times 10^6$ (K cal/day)

PbS:

$H_{958} - H_{298°K} = +8.66$ (K cal/mole)

Total heat $= +8.66 \times 1.561 \times 10^5 = +1.352 \times 10^6$ (K cal/day)

FeS_2:

$H_{958} - H_{298°K} = +12.35$ (K cal/mole)

Total Heat $= +12.35 \times 2.9350 \times 10^6 = +3.6247 \times 10^7$ (K cal/day)

Total heat necessary to preheat the feed:

ΔH_9 = +8.116 x 10^7 (K cal/day)

.4 Reheat Recirculated Dust

Assuming, for simplification(*), that dust contains only $ZnSO_4$, Fe_2O_3, $PbSO_4$ and $CuSO_4$. Dust return to reactor at 600°C.

The dust elutriation flux from the reactors is:

K^* = 1.038 x 10^{-4} (T/M^2 x sec) (685°C, u_o' = 75 cm/sec)
k^* = 980.77 TPD dust recirculated

$ZnSO_4$:
$H_{958} - H_{873°K}$ = +3.28 (K cal/mole)
Total heat = +3.28 x 5.026 x 10^6 = +1.649 x 10^7 (K cal/day)

Fe_2O_3:
$H_{958} - H_{873°K}$ = +0.85 (K cal/mole)
Total heat = +0.85 x 1.7195 x 10^6 = +1.462 x 10^6 (K cal/day)

$PbSO_4$:
$H_{958} - H_{873°K}$ = +3.27 (K cal/mole)
Total heat = +3.27 x 1.559 x 10^5 = +5.098 x 10^5 (K cal/day)

$CuSO_4$:
$H_{958} - H_{873°K}$ = +3.23 (K cal/mole)
Total heat = +3.23 x 0.962 x 10^5 = +3.107 x 10^5 (K cal/day)

Total heat required to reheat the dust:

ΔH_{10} = +1.877 x 10^7 (K cal/day)

(*) The total heat required to reheat the dust is very small [illegible]

.5 Vaporize and Decompose HAL Solution

Values for the heat of formation and heat capacities are given in Table A3-2.

TABLE A3-2

STANDARD HEAT OF FORMATION AND HEAT CAPACITIES OF HAL SOLUTION

Compound	$\Delta H°_{298°K}$ (K cal/mole)	C_p (cal/mole-°K)
Fe_2O_3	-196.3	$23.49 + 16.6 \times 10^{-3}$ T
$FeSO_4$	-221.3	22.0
$Fe_2(SO_4)_3$	-617.0	66.2
$CuSO_4$	-184.0	$18.77 + 17.20 \times 10^{-3}$ T
$ZnSO_4$	-234.9	$21.90 + 18.2 \times 10^{-3}$ T
SO_2 (g)	- 71.0	$10.38 + 2.54 \times 10^{-3}$ T
H_2SO_4	-194.6	33.2
O_2	-	$7.16 + 1.0 \times 10^{-3}$ T
H_2O (g)	- 68.3	$7.30 + 2.46 \times 10^{-3}$ T

HAL solution composition (main constituents):

$ZnSO_4$ = 112.78 TPD = 6.986×10^5 (moles/day)
$FeSO_4$ = 18.74 " = 1.234×10^5 "
$Fe_2(SO_4)_3$ = 177.71 " = 4.444×10^5 "
H_2SO_4 = 21.06 " = 2.148×10^5 "
H_2O = 472.76 " = 2.626×10^7 "

803.05 TPD

.51 Preheat Dissolved Solids and H_2SO_4 in HAL Solution

HAL solution is recirculated back into the roaster at 90°C

$$ZnSO_4 = 21.9\ (958 - 363) + 9.1 \times 10^{-3}\ (\overline{958}^2 - \overline{363}^2)$$
$$= 20.18 \text{ (K cal/mole)}$$

$FeSO_4$ = 22.0 (958 - 363) = 13.09 (K cal/mole)

$Fe_2(SO_4)_3$ = 66.2 (958 - 363) = 39.39 (K cal/mole)

H_2SO_4 = 33.2 (958 - 363) = 12.05 (K cal/mole)

Therefore,

$ZnSO_4 = 20.18 \times 6.986 \times 10^5 = 1.410 \times 10^7$ (K cal/day)
$FeSO_4 = 13.09 \times 1.234 \times 10^5 = 1.615 \times 10^6$ "
$Fe_2(SO_4)_3 = 39.39 \times 4.444 \times 10^5 = 1.750 \times 10^7$ "
$H_2SO_4 = 12.05 \times 2.148 \times 10^5 = 2.588 \times 10^6$ "

Total to preheat dissolved solids in HAL solution:

$\Delta H_{11} = +3.581 \times 10^7$ (K cal/day)

.52 Decompose HAL Solution (no water)

$ZnSO_4$ remain as such.

$FeSO_4$:
$FeSO_4 \xrightarrow{\Delta} 0.5\ Fe_2O_3 + SO_2 + 0.25\ O_2$

$\Delta H^\circ_{298^\circ K} = +52.15$ (K cal)
$\Delta C_p = 3.71 + 11.34 \times 10^{-3}\ T$ (cal/mole °K)
$\Delta H_o = +50{,}541$ (cal)

$\Delta H^\circ_T = 50{,}541 + 3.71T + 5.67 \times 10^{-7}$ (K cal)
$\Delta H^\circ_{958^\circ K} = +59.30$ (K cal)

$Fe_2(SO_4)_3$:
$Fe_2(SO_4)_3 \xrightarrow{\Delta} Fe_2O_3 + 3SO_2 + 1.5O_2$

$\Delta H^\circ_{298^\circ K} = +207.7$ (K cal)
$\Delta C_p = -0.83 + 25.7 \times 10^{-3}\ T$ (cal/mole °K)
$\Delta H_o = +206{,}801$ (cal)

ΔH°_T = +206,801 - 0.83T + 12.9 x 10^{-3} T (K cal)
$\Delta H^\circ_{958^\circ K}$ = +217.85 (K cal)

H_2SO_4:
$H_2SO_4 \underset{\Delta}{\to} H_2O + SO_2 + 0.5O_2$.

$\Delta H^\circ_{298^\circ K}$ = +55.3 (K cal)
ΔC_p = -12.07 + 5.6 x 10^{-3} T (cal/mole °K)
ΔH_o = +58,648 (cal)

ΔH°_T = +58,648 - 12.07T + 2.8 x 10^{-3} T^2 (K cal)
$\Delta H^\circ_{958^\circ K}$ = +49.66 (K cal)

Therefore heat to decompose each component is:

$FeSO_4$ = 59.30 x 1.234 x 10^5 = +7.318 x 10^6 (K cal/day)
$Fe_2(SO_4)_3$ = 217.85 x 4.444 x 10^5 = 9.6810 x 10^7 "
H_2SO_4 = 49.66 x 2.148 x 10^5 = +1.067 x 10^7 "

Total to decompose solids and H_2SO_4 in HAL solution:

ΔH_{12} = +1.1480 x 10^8 (K cal/day)

A3.5 OVERALL HEAT BALANCE

.51 Heat Generated (ΔH_R)

By chemical reactions:

ΔH_R = -1.5014 x 10^9 (K cal/day)

.52 Heat Consumed (ΔH_C)

By heat losses through reactor shell:

ΔH_7 = +7.507 x 10^7 (K cal/day)

To preheat the fluidizing air:

$\Delta H_8 = +6.8346 \times 10^8$ (K cal/day)

To preheat the feed:

$\Delta H_9 = +8.116 \times 10^7$ (K cal/day)

To reheat recirculated dust:

$\Delta H_{10} = +1.877 \times 10^7$ (K cal/day)

To preheat HAL solution:

$\Delta H_{11} = +3.581 \times 10^7$ (Kcal/day)

To decompose HAL solution:

$\Delta H_{12} = +1.1480 \times 10^8$ (K cal/day)

.53 Excess Heat to Vaporize H_2O

$\Delta H_E = \Delta H_R - \Delta H_C = -4.923 \times 10^8$ (K cal/day)

Water to vaporize:

$H_2O = (-4.923 \times 10^8/-16.326) \times 18/10^6$

$H_2O = 542.8$ TPD

(total H_2O in HAL solution from tailings refloat products is 472.76).

Therefore, the reactor has an extra capacity to consume 70.1 TPD equivalent water.

.54 Spent Additional to Consume

Assuming feed contains 2.5% moisture, heat to vaporize this water is:

$$\Delta H \text{ moisture} = (22.51 \times 10^6/18) \times 16.326$$
$$= +\ 2.04 \times 10^7 \text{ (K cal/day)}$$

(The total net heat surplus allows to use a feed with up to 7.8% moisture).

Net surplus after vaporizing 2.5% moisture:

$$\Delta H \text{ surplus} = +4.349 \times 10^7 \text{ (K cal/day)}$$

Spent electrolyte of 60 gpl Zn, 165 gpl H_2SO_4 consumes 9.84×10^5 (K cal/Tm spent).

$$\text{Total spent additional} = 4.349 \times 10^7/9.84 \times 10^5$$
$$= 44.20 \text{ TPD}$$

The total consumption of heat by the HAL solution and spent electrolyte represents the net amount of heat to be eliminated from the fluidized bed alone, not considering heat recovered from the hot gases above the fluidized bed level. In practice, since the spray gun for the HAL solution and spent are located above the bed level, some heat is recovered from the hot gases. This will increase the overall capacity of the reactor for consuming HAL solution and spent, as is discussed in detail in Appendix A1.

A3.6 SOURCES OF THERMODYNAMICAL DATA

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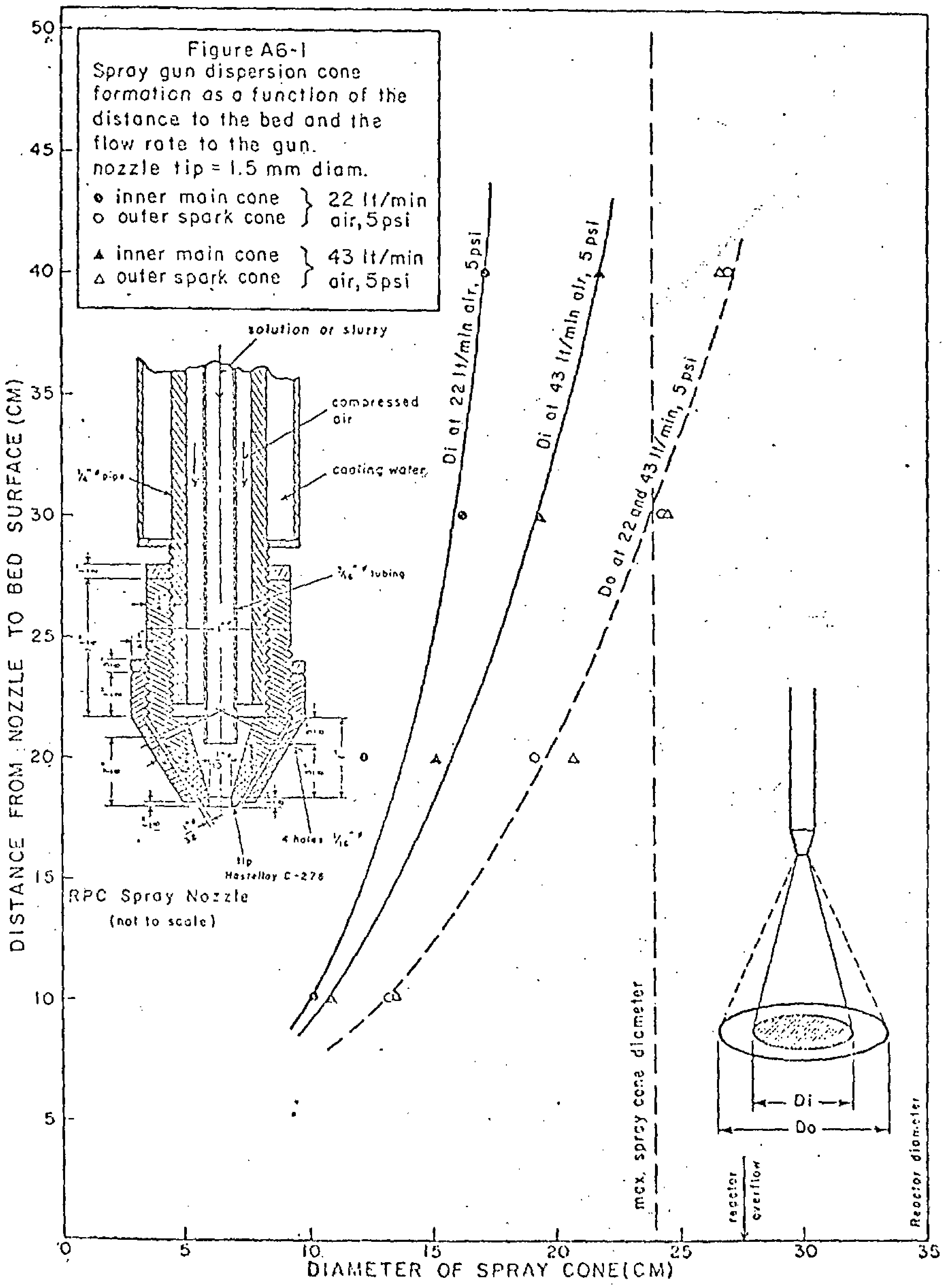


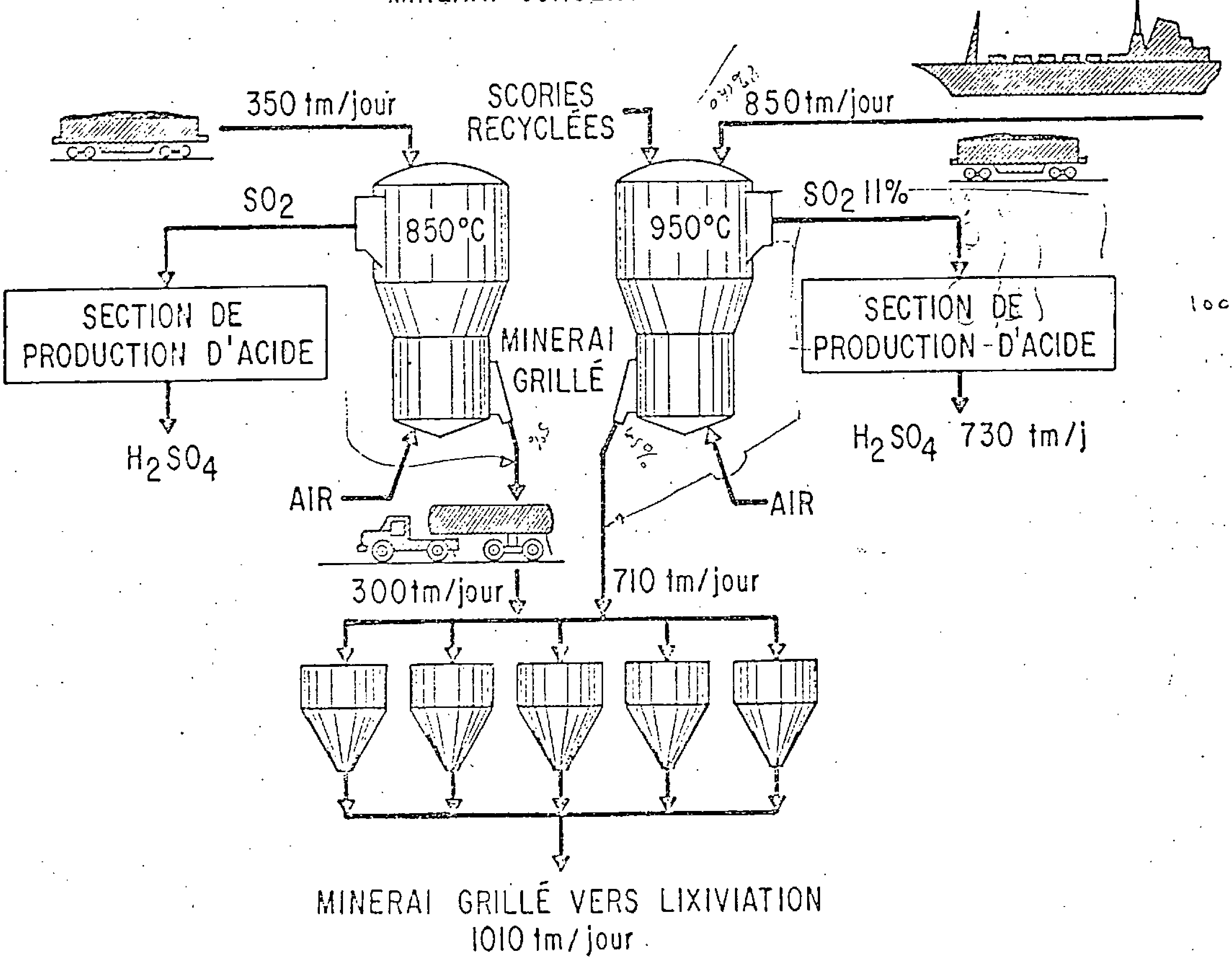
Figure A6-1
Spray gun dispersion cone formation as a function of the distance to the bed and the flow rate to the gun.
nozzle tip = 1.5 mm diam.

ANNEX 2

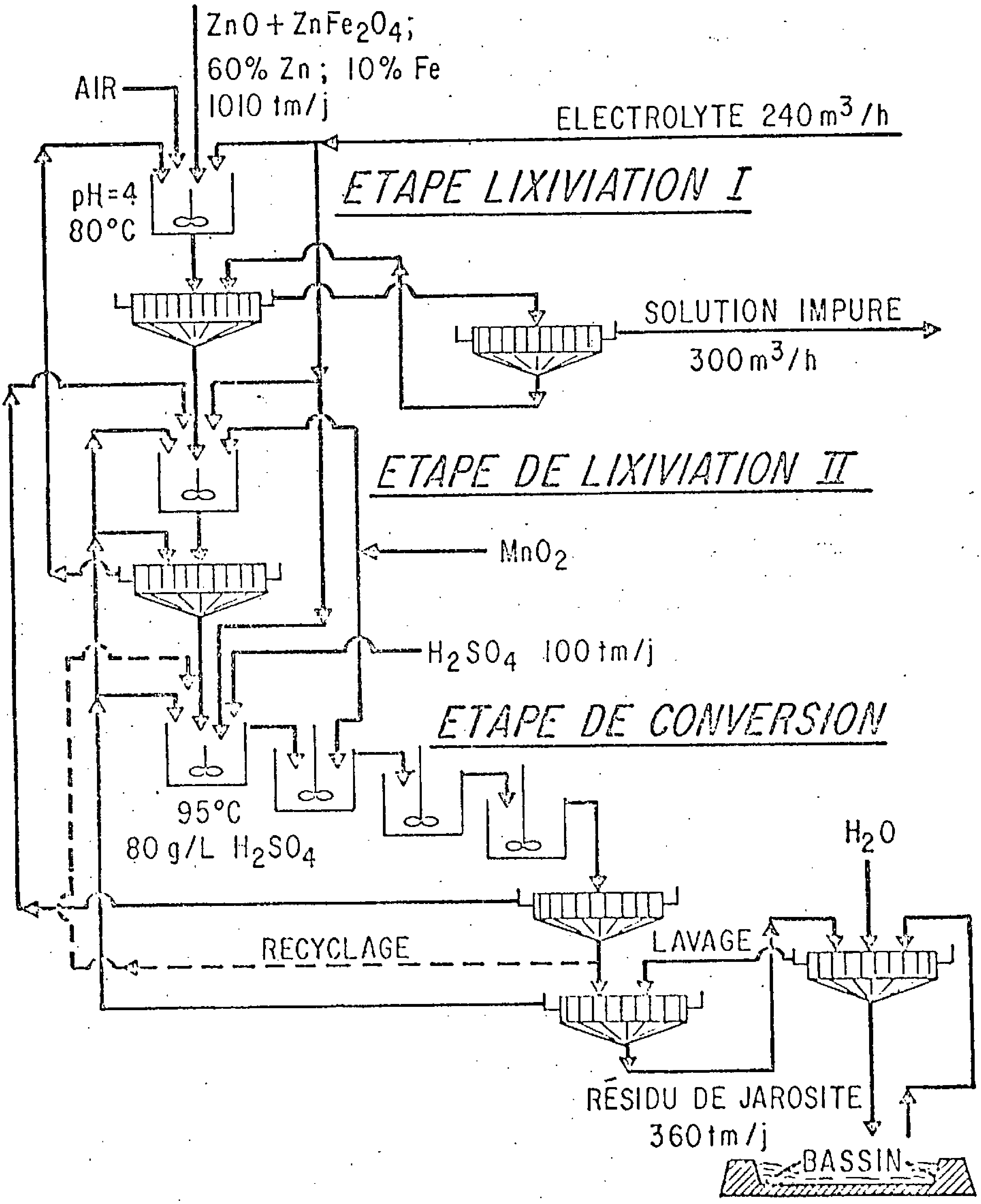
Canadian Electrolytic Zinc, C.E.Z.

GRILLAGE

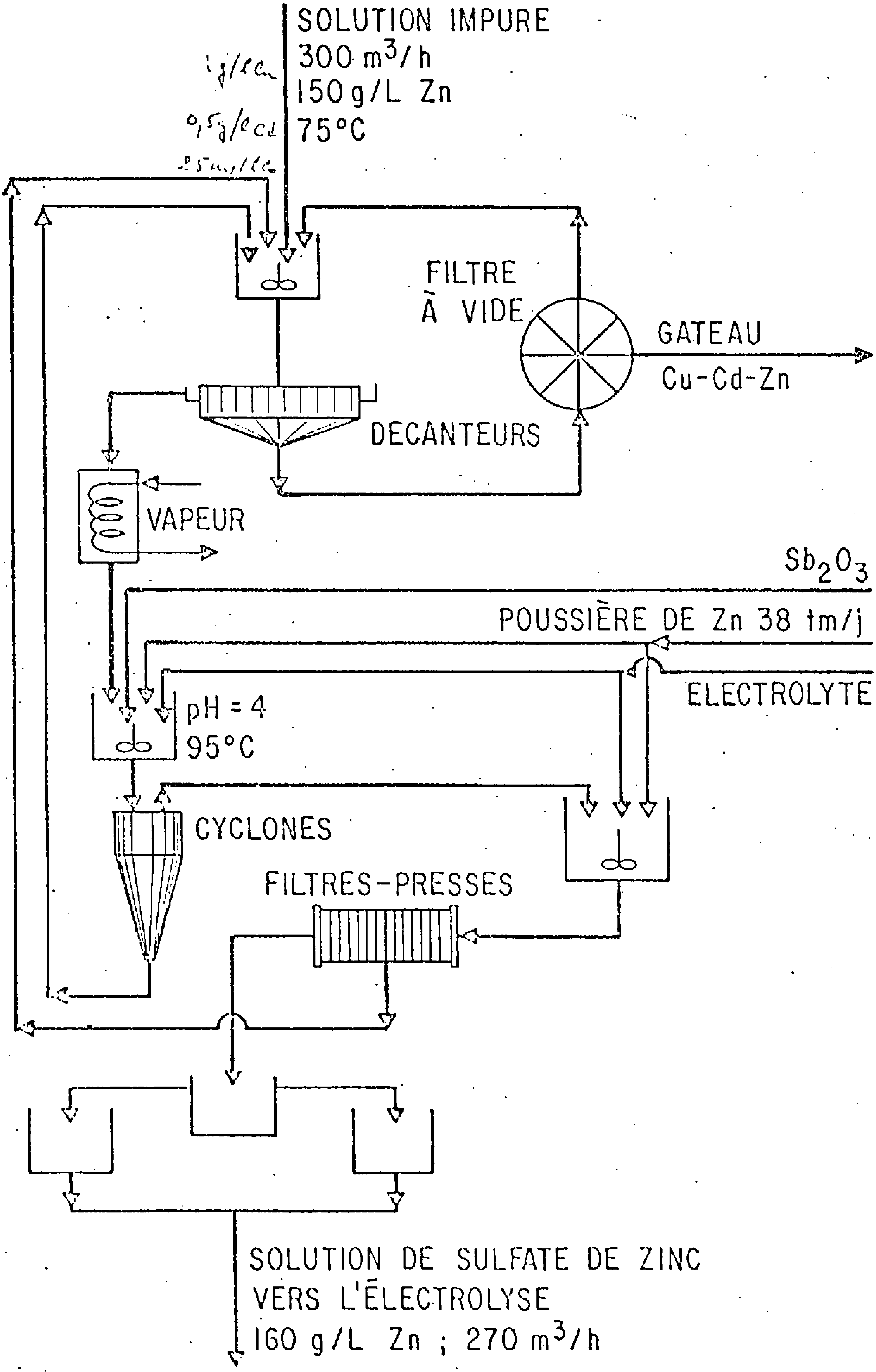
MINERAI CONCENTRÉ : ZnS 53% Zn



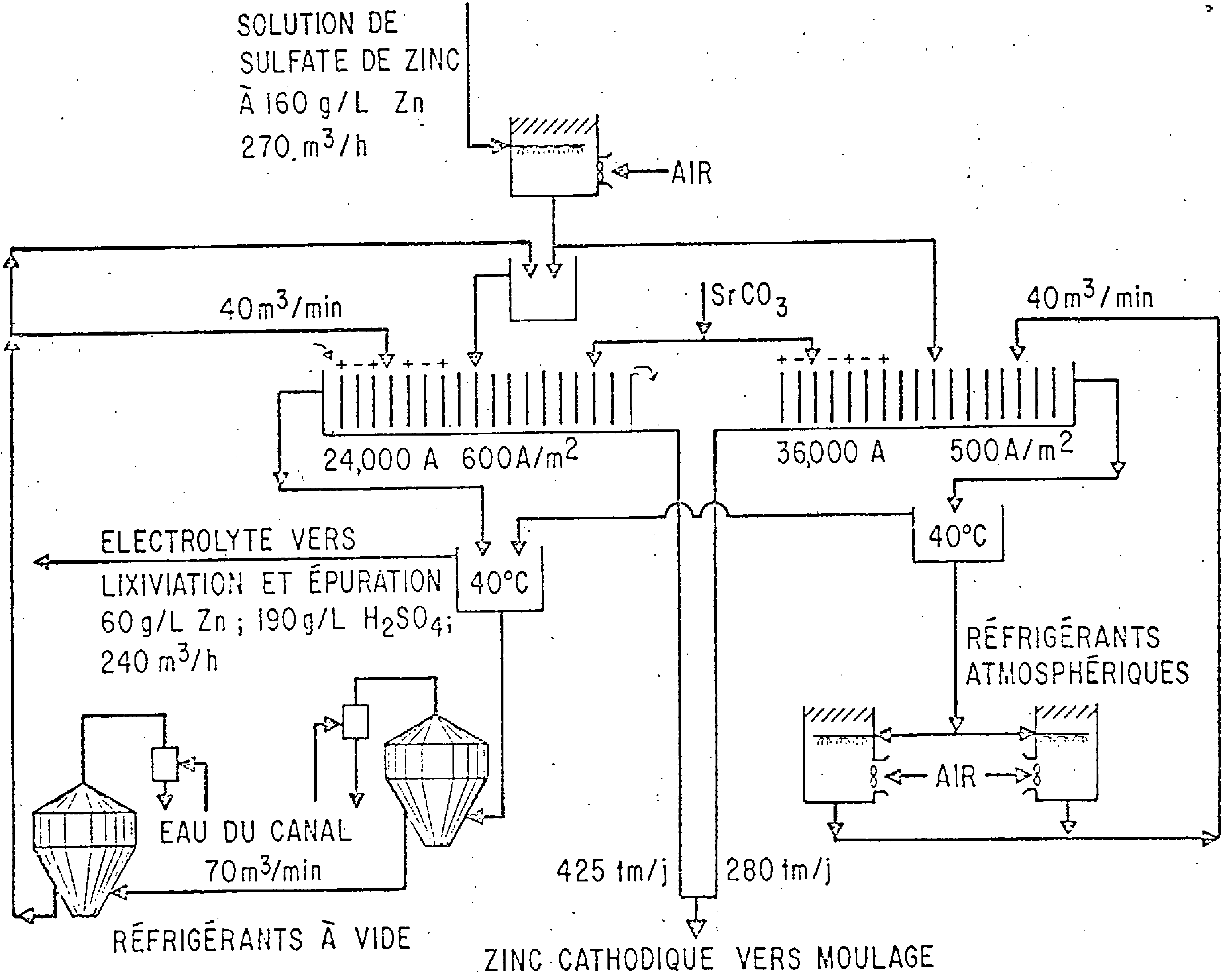
LIXIVIATION



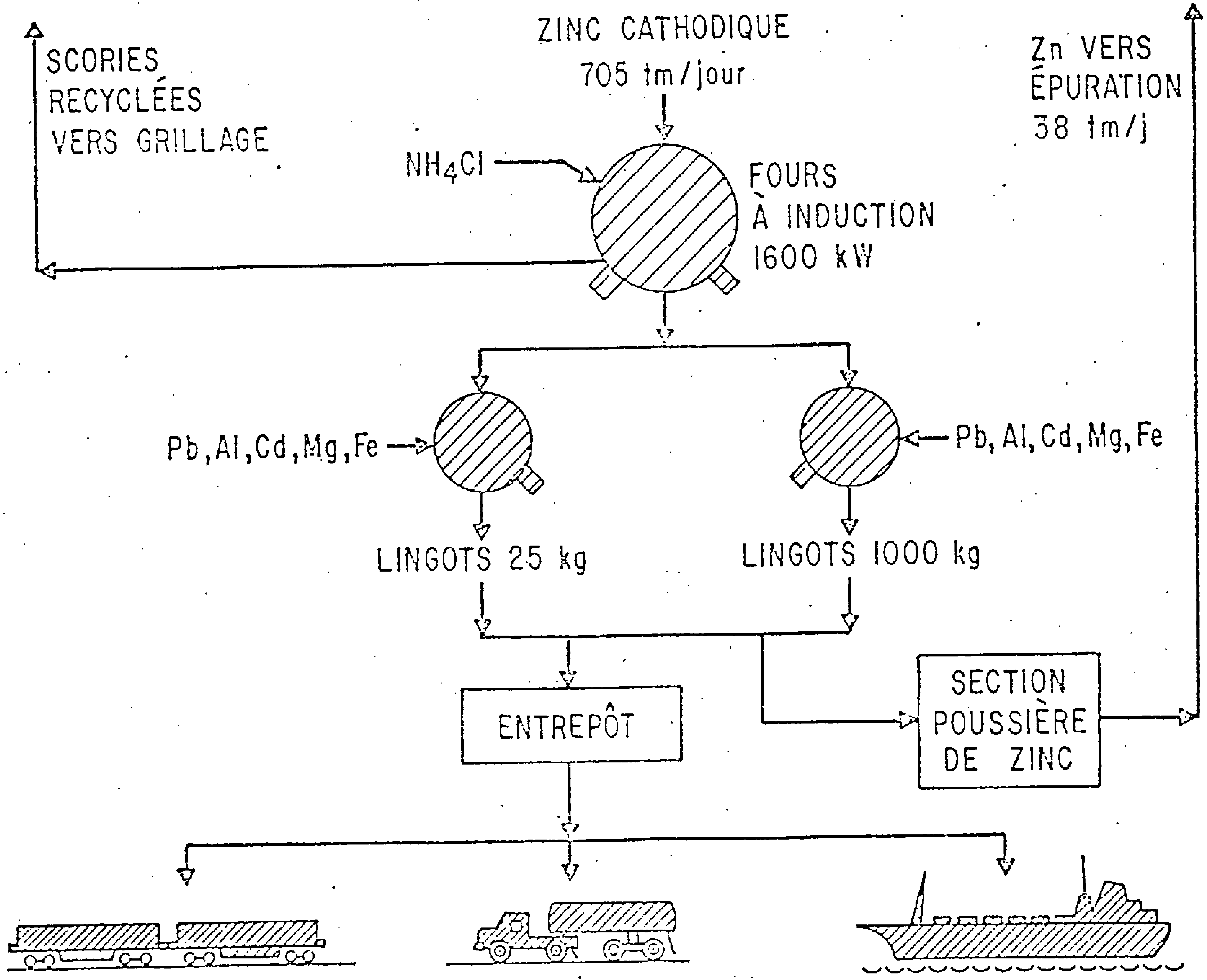
EPURATION


SOLUTION IMPURE
300 m³/h
150 g/L Zn
75°C
1 g/l Cu
0,5 g/l Cd
25 mg/l Co
FILTRE À VIDE
GATEAU
Cu-Cd-Zn
DECANTEURS
VAPEUR
Sb₂O₃
POUSSIÈRE DE Zn 38 tm/j
ELECTROLYTE
pH = 4
95°C
CYCLONES
FILTRES-PRESSES
SOLUTION DE SULFATE DE ZINC
VERS L'ÉLECTROLYSE
160 g/L Zn ; 270 m³/h

ELECTROLYSE



MOULAGE



EXPEDITION : 630 tm/jour DE LINGOTS

Opérations de grillage des minerais de zinc en lit fluidisé

par P. Duhaime et P. Krick

Zinc Electrolytique du Canada Limitée

A. INTRODUCTION

Le terme "grillage" fait référence à des réactions gaz-solide à température élevée, lors du traitement de minerais concentrés pour la récupération des métaux.

Le grillage à l'échelle industrielle peut se faire dans des fours rotatifs, fours à soles multiples et fours à lit fluidisé.

Les systèmes gaz-solide entrant en jeu dans le grillage ne sont pas faciles à comprendre, tant du point de vue chimique que du point de vue thermodynamique et cinétique. L'on peut dire, sans trop se tromper, qu'aujourd'hui encore l'art du grillage est en avance sur la science.

Le point le plus important dans le choix d'un type de four est le coût de l'investissement et le coût d'opération par tonne de minerai traité. Ceci veut dire qu'il faut de grandes vitesses de réaction, un transfert de chaleur maximum, de gros fours pour de gros tonnages et un contrôle fiable et précis ne requérant que peu de main-d'oeuvre.

C'est parce que les fours à lit fluidisé réunissent ces caractéristiques que la plupart des producteurs de zinc les ont adoptés. Le plus grand en opération dans le monde en 1980, du type Lurgi, a une sole de 125 m^2 et peut griller 800 t/j de minerai concentré. Il se trouve en Australie.

B. MODES DE GRILLAGE

1) Grillage à mort: type Métallurgie Hoboken-Overpelt

Le minerai concentré est aggloméré artificiellement par addition d'un peu d'H_2SO_4, granulé et séché. A peu près tous les solides restent dans le lit, et seule une petite fraction est entraînée vers les cyclones. Le minerai grillé, formé de ZnO et de $ZnFe_2O_4$, est évacué par le trop-plein et refroidi. Ce type de grillage a la propriété de donner du soufre résiduel total de l'ordre de 0.3% et de traiter des blendes pauvres sans que le cuivre ou le plomb n'interfèrent. Capacité spécifique: 1.1 t/h/m^2, température: 1000°C.

2) Grillage à mort: type Lurgi

Dans ce mode, environ 40% des solides sortent par le trop-plein du four. Les autres 60% s'échappent avec les gaz par le haut du four et sont collectés dans le récupérateur de chaleur, les cyclones et le dépoussiéreur électrostatique. Le soufre résiduel total dans ce cas est d'environ 2%. Le minerai grillé est comme précédemment formé de ZnO et de $ZnFe_2O_4$. Capacité spécifique: 0.3 t/h/m^2, température 850-1000°C.

.../2

3) Grillage sulfatant: procédé RPC*

Le minerai subit un grillage sulfatant à 630°C dont le principe est de former des sulfates et oxysulfates de zinc au lieu d'oxydes. Le Fe est transformé en Fe_2O_3 insoluble. L'avantage est une lixiviation très facile. L'inconvénient est l'élimination ultérieure des sulfates. Ce procédé est actuellement à l'essai à petite échelle afin de prouver son application industrielle.

C. EQUIPEMENT DE BASE POUR UNE INSTALLATION DE GRILLAGE

1) Hangar de dégel, hangar de déchargement, hangar d'entreposage et de mélange, silos.

2) Mélangeurs, granuleurs, balance, injecteurs pour le minerai concentré.

3) Souffleur, débitmètre pour l'injection d'air.

4) Four à lit fluidisé en milieu turbulent.

5) Serpentins de refroidissement incorporés dans le lit et récupérateurs de chaleur à la sortie du four. Ceci représente la plus grosse source de chaleur pour toute l'usine.

6) Système de séparation solide-gaz: chambre de sédimentation, cyclones et dépoussiéreur électrostatique.

7) Système de classification: broyeur, classificateur, cyclones, dépoussiéreur à manches.

8) Système de transport pneumatique vers la section lixiviation.

D. GRILLAGE DES CONCENTRES DE ZINC

Les concentrés de zinc, appelés également blendes, sont en général obtenus par flottation. Ces concentrés peuvent être très fins, comme par exemple ceux du Nouveau-Brunswick qui ont une granulométrie à 85% inférieure à 44 µ. Ceci provient du fait que le grain de sphalérite dans le minerai est lui-même très petit, et que pour l'isoler pour fin de flottation il faut le broyer finement.

1) Composition chimique des blendes

Le tableau 1 donne l'analyse de trois (3) types de minerais.

---

*Le Conseil de Recherche et de Productivité (N-B)

Tableau I

Elément	Minerai concentré		
	Mattagami %	BM & S %	Balmat %
Zn	52	52	60
S	33	33	32
Fe	10.6	9.4	3.7
Cu	0.65	0.30	0.09
Cd	0.13	0.09	0.14
Mg	0.03	0.08	0.20
Mn	0.21	0.06	0.13
Co	0.008	0.007	0.003
Cl	0.001	0.006	0.010
Hg	0.0005	0.0018	0.0360
As	0.007	0.13	0.008
Pb	0.13	1.6	0.5
Ag	0.0040	0.0080	0.0015
H_2O	4.7	6.9	5.2

Les minerais contiennent toujours de petites quantités de métaux qui seront récupérés ou éliminés ultérieurement. Les halogènes, l'arsenic, le mercure, le sélenium, quant à eux, sont volatilisés et sont éliminés des gaz avant la production d'acide sulfurique.

2) Composition minéralogique des blendes

Le sulfure de zinc forme en général une solution solide avec du sulfure de fer. Ces minéraux isomorphes sont connus sous l'appellation de marmatites. Le rapport molaire Zn/Fe varie de 2 à 5.

.../ 4

Le tableau II donne la composition minéralogique de deux (2) types de minerais.

Tableau II

Composé		Minerai Mattagami %	Minerai Geco %
sphalerite	ZnS	76	73
pyrrhotite	$Fe_{1-x}S$	18	17
pyrite	FeS_2	0	4
galène	PbS	Traces	Traces
hématite	Fe_2O_3	0	0
magnetite	Fe_3O_4	0	0
restant		6	6

3) Réactions chimiques et thermodynamiques

Le grillage est un procédé complexe, non pas à cause des réactions prises individuellement, mais bien plus par le fait qu'elles peuvent se produire simultanément et que des interactions peuvent en résulter.

La thermodynamique demeure le meilleur outil pour étudier les réactions. Auparavant, dans le grillage à sole fixe, les conditions du grillage variaient jusqu'à l'infini dépendant du point où l'on se trouvait. Le grillage se faisait alors dans des conditions hors-équilibre, et, pour ce type de grillage, la thermodynamique n'était pas très utile. Aujourd'hui avec le grillage à lit fluidisé les conditions de grillage sont très homogènes à travers le lit, et on tend beaucoup plus vers l'équilibre.

La figure 1 montre les composés possibles dans le système quaternaire Zn-Fe-S-O. Les points intéressants à noter dans ce système sont:

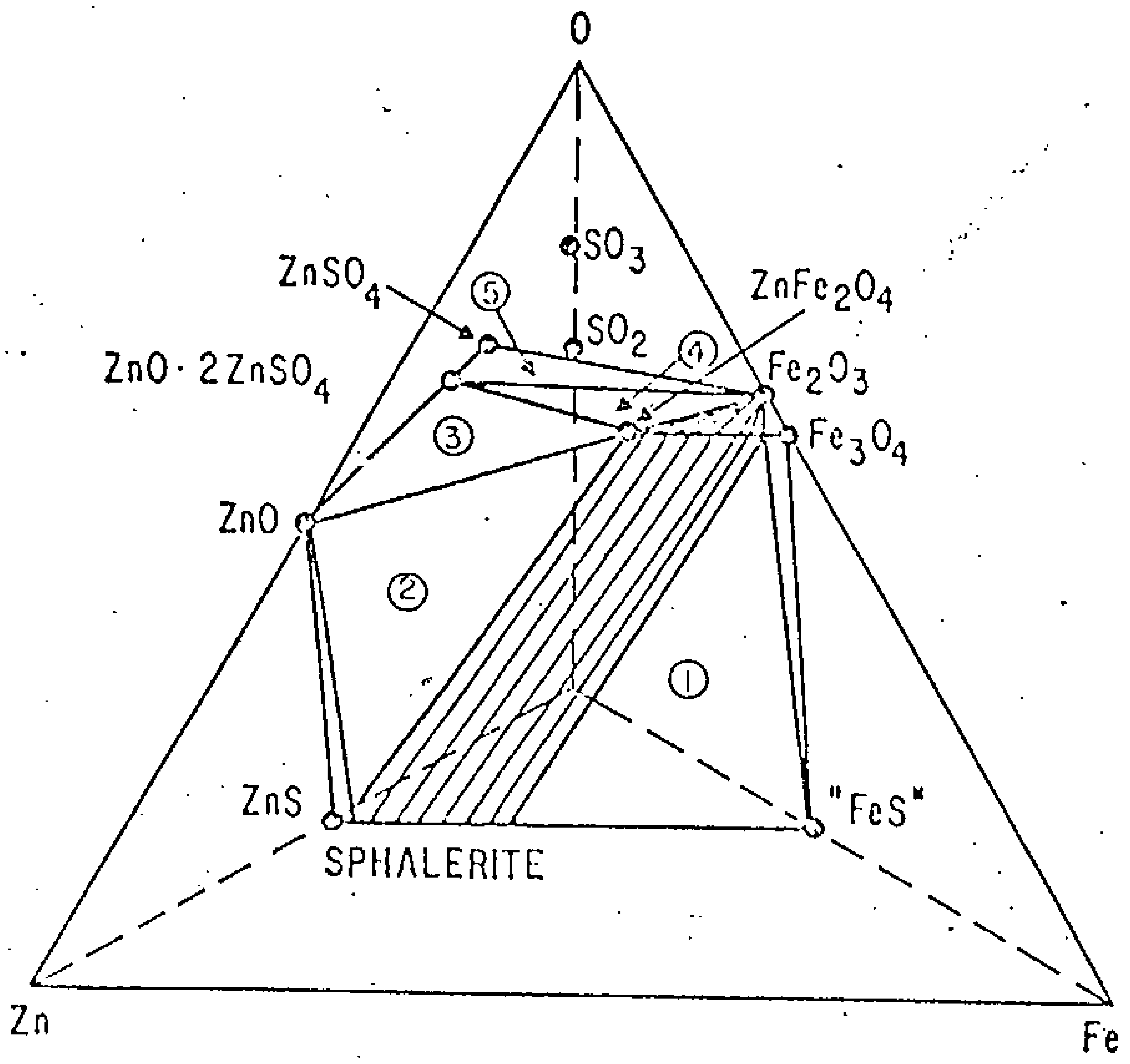
a) possibilité pour le ZnS et FeS de former un composé $(Zn, Fe)S$ appelé sphalérite ayant différentes teneurs de FeS;

b) le même phénomène se produit pour le $ZnFe_2O_4$ et Fe_3O_4. Il est donc possible d'avoir un composé de $ZnFe_2O_4 - Fe_3O_4$ n'ayant pratiquement pas de zinc. Les autres composés rencontrés sont le ZnS, ZnO, $ZnO \cdot 2\,ZnSO_4$, Fe_2O_3, FeS, SO_2 et SO_3. Ceci est vrai à 900-950°C et 1 atm de SO_2.

La réaction principale dans le grillage du zinc est l'oxydation du sulphure de zinc.

.../ 5

Figure I



SYSTEME Zn-Fe-S-O À 1 ATM. SO_2 ET 900°C

(1) $ZnS + 3/2\ O_2 \longrightarrow ZnO + SO_2$

$\Delta G° = -107595 + 17.16\ T$ pour T > 1180°K

A 1200°K par exemple, pour un P_{SO_2} de 0,1 atm le PO_2 correspondant est 1.16 10^{-12}.

Les autres réactions possibles sont:

Réaction	T°K	PSO_2 atm	PO_2 atm
(2) $ZnO + SO_2 + 1/2\ O_2 \longrightarrow ZnSO_4$	1200	0.1	7.94
(3) $ZnS + 2\ O_2 \longrightarrow ZnSO_4$	1200	0.1	5.95 10^{-9}

(4) $3\ FeS + 5\ O_2 \rightleftarrows Fe_3O_4 + 3\ SO_2$	1200	0.1	$7.6\ 10^{-11}$
(5) $2\ FeS + 7/2\ O_2 \rightleftarrows Fe_2O_3 + 2\ SO_2$	1200	0.1	$1.93\ 10^{-16}$
(6) $2\ FeS + 5/2\ O_2 + SO_2 \rightleftarrows Fe_2(SO_4)_3$	1200	0.1	$1.11\ 10^{-13}$
(7) $Fe_2O_3 + 1/2\ O_2 + 2\ SO_2 \rightarrow 2\ FeSO_4$	1200	0.1	$1.06\ 10^{7}$

La réaction (2) ne peut se produire car le PO_2 à l'intérieur du four est d'environ 0.03 atm. La réaction (3) est possible, mais le ZnS a plus tendance à se décomposer en ZnO car le PO_2 pour la réaction (1) est plus petit que pour la réaction (3). C'est donc la réaction (1) qui va se produire. Nous voyons que c'est la réaction (5) qui se produira le plus en ce qui regarde le fer. Il n'y aura pas de $FeSO_4$ de formé à 1200°K.

La thermodynamique prédit donc que nous aurons du Fe_2O_3 et du ZnO à 1200°K avec PSO_2 égal à 0.1 atm. Il y a par contre une autre réaction possible entre le ZnO et le Fe_2O_3 avec formation de ferrite de zinc $ZnFe_2O_4$.

(8) $ZnO + Fe_2O_3 \longrightarrow ZnFe_2O_4$

$\Delta G^o = -1120 + 0.90\ T$

pour $P_{SO_2} = 1$ atm $\qquad P_{O_2} = 3\ 10^{-11}$ atm.

La tendance du ZnO et de Fe_2O_3 à réagir l'un avec l'autre est donc très forte.

Des graphiques ont été établis afin de représenter sur une même figure les différents paramètres thermodynamiques.

Il est intéressant de suivre la séquence des réactions sur la figure II:

i) A 950°C (8.2 sur le graphe) jusqu'à log P_{O_2} égal à -11, le ZnS et le FeS sont stables.

ii) Entre -11 et -9.5 il se forme une ferrite non stoechiométrique avec le ZnO et le Fe_2O_3.

iii) A -10.5 le ZnO est stable avec du $ZnFe_2O_4$. Ce dernier composé est stable sous les conditions rencontrées dans le grilleur, c'est-à-dire à P_{O_2} égal à 0.03 atm. Il faut noter que la pression d'oxygène qui compte n'est pas celle qui existe dans le four, mais plutôt celle se trouvant à l'intérieur de la particule de ZnO-ZnS. Cette pression est normalement plus faible que celle du four.

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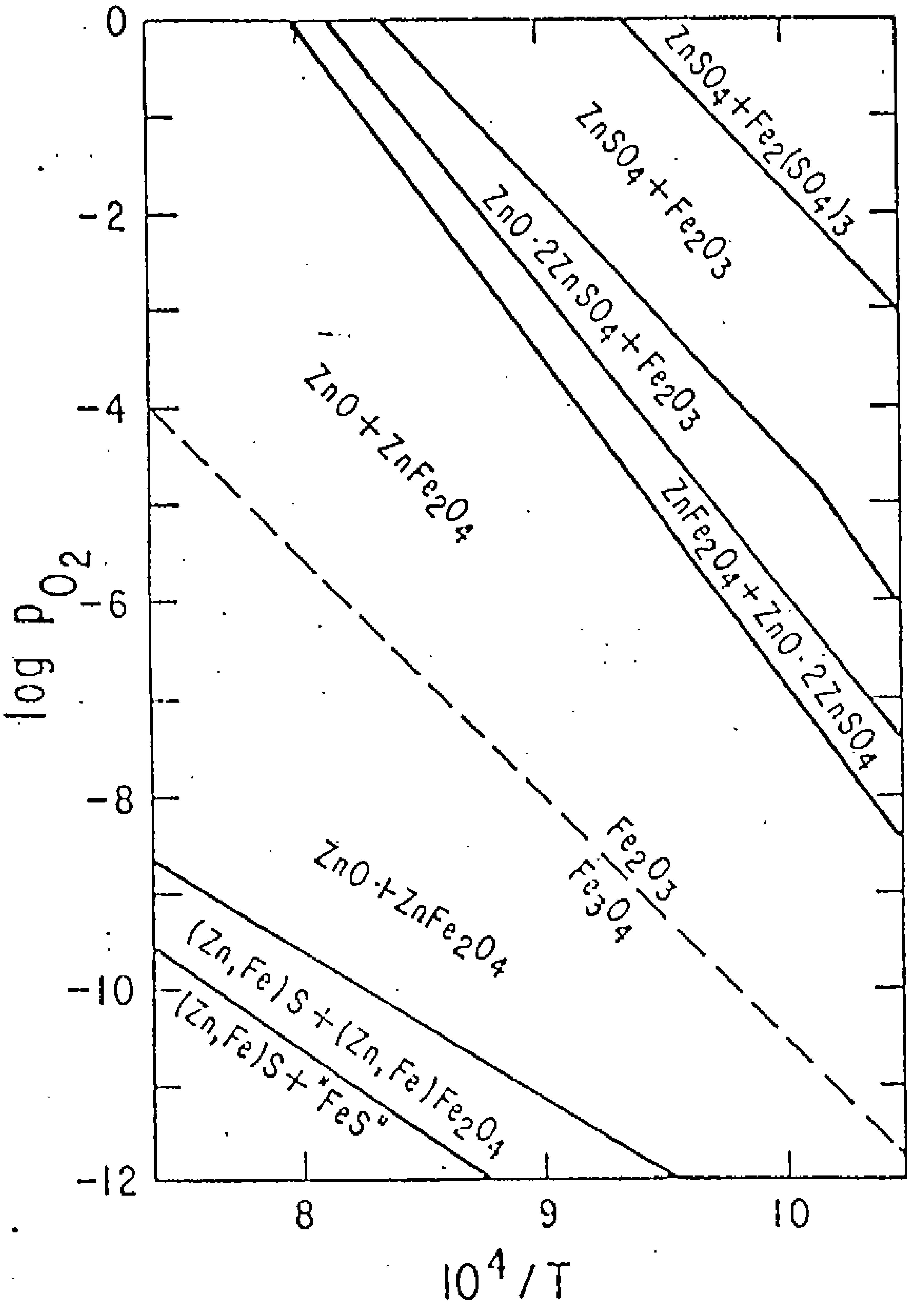
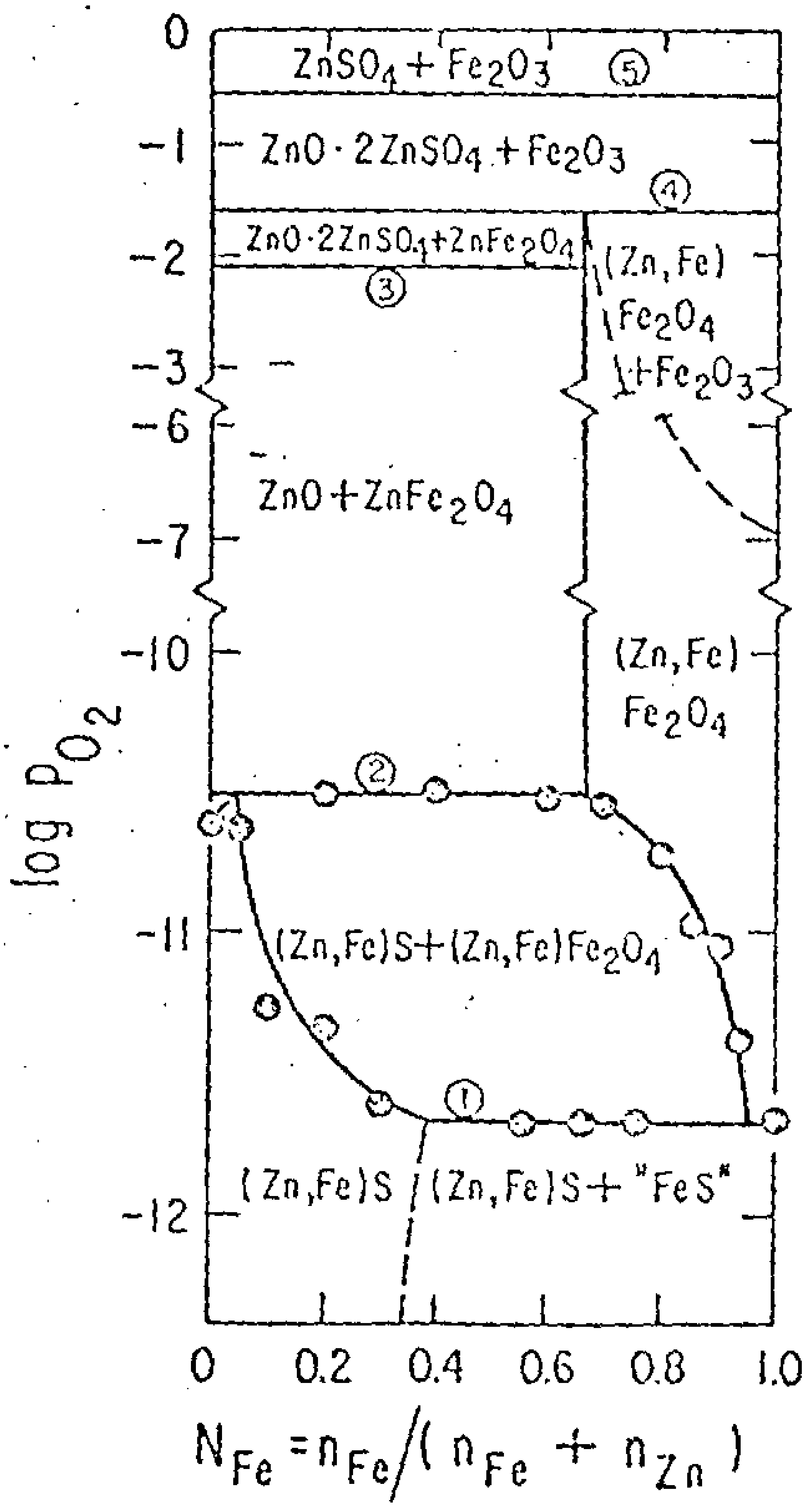
Figure II



POTENTIEL O_2 EN FONCTION DE LA TEMPERATURE
POUR LE SYSTEME Zn-Fe-S-O (pSO_2 = 1 atm)

Figure III



POTENTIEL O_2 EN FONCTION DE LA FRACTION MOLAIRE DU Fe DANS LE SYSTEM Zn-Fe-S-O ($p\ SO_2$ = 1 atm, 890°C)

La figure III montre la variation de composition des différents composés, principalement la ferrite, à température constante de 890°C et pression partielle de SO_2 constante à 1.0 atm. Le fait que nous ayons une pression moindre de SO_2 dans le four (0.1 atm) a comme influence d'abaisser la pression partielle d'équilibre de l'oxygène. Notre sphalerite a un rapport N_{Fe} d'environ 0.1. Le premier (Zn,Fe)Fe_2O_4 à se former [illegible] une teneur en zinc d'environ 10% et le dernier à se former, à log P_{O_2} égal à -10.5, en aura une de 35%. Ensuite il n'y aura plus de variation dans la composition et le produit final sera comme on l'a déjà prévu, composé de ZnO et de $ZnFe_2O_4$. En pratique, nous savons que tout notre fer dans le minerai grillé est combiné avec du ZnO pour former des ferrites, qui sont faiblement magnétiques.

La thermodynamique nous a aussi montré que, dans le four, il n'y avait pratiquement pas de sulphate de formé. L'analyse du minerai grillé en indique, par contre. Il est formé lors du refroidissement dans le récupérateur de chaleur, les cyclones et le dépoussiéreur électrostatique.

L'étude thermodynamique des ferrites est importante, car elle permet de connaître les conditions nécessaires pour éviter leur formation. Il est actuellement possible de dire, qu'avec une pression partielle d'oxygène élevée ($>$ 0.1 atm) on peut éviter leur formation à 900°C. Une autre possibilité est de griller à basse température (680°C), et ainsi former des sulphates au lieu d'oxydes (procédé RPC).

4) Cinétique des réactions

Le four à lit fluidisé est caractérisé par des coefficients de transfert de chaleur et de masse très élevés. La turbulence dans le lit, la vitesse relative élevée du gaz, de l'ordre de 0.6 m/s et le contact gaz-solide très intime en sont les causes. Des essais ont démontré que la vitesse d'oxydation dans un four à lit fluidisé est de 1.5 à 2 fois plus rapide que dans un four du type sole fixe. Le transfert de chaleur y est jusqu'à 4 fois plus rapide.

L'étude de la cinétique de la réaction d'oxydation d'une sphalérite pure a démontré que la vitesse augmente rapidement au début pour ensuite ralentir. L'explication de ce phénomène est liée au fait que, au début, la vitesse est contrôlée par la réaction de chimisorption (jusqu'à 830°C) et que, par la suite, c'est la diffusion du gaz SO_2 à travers la couche d'oxyde qui la contrôle. Le modèle généralisé pour l'oxydation d'une sphère de ZnS est montré sur la figure IV.

On cherchera donc, au point de vue pratique, à avoir la température la plus élevée possible (950°C), la vitesse des gaz maximale, des particules fines et une teneur élevée en oxygène. Par contre, à cause de l'équilibre SO_2/SO_3 on minimisera cet excès. Le SO_3 avec la vapeur d'eau donne H_2SO_4 qui corrode l'équipement. On sait que l'eau ajoutée au concentré agit aussi comme agent agglomérant, c'est-à-dire favorise la présence de grosses particules; pour éviter une trop forte proportion de granules, on essaiera de ne pas trop en ajouter.

D'autre part, une teneur en fer d'environ 10% dans le minerai concentré ralentit la vitesse de réaction à cause du rôle agglomérant du FeS.

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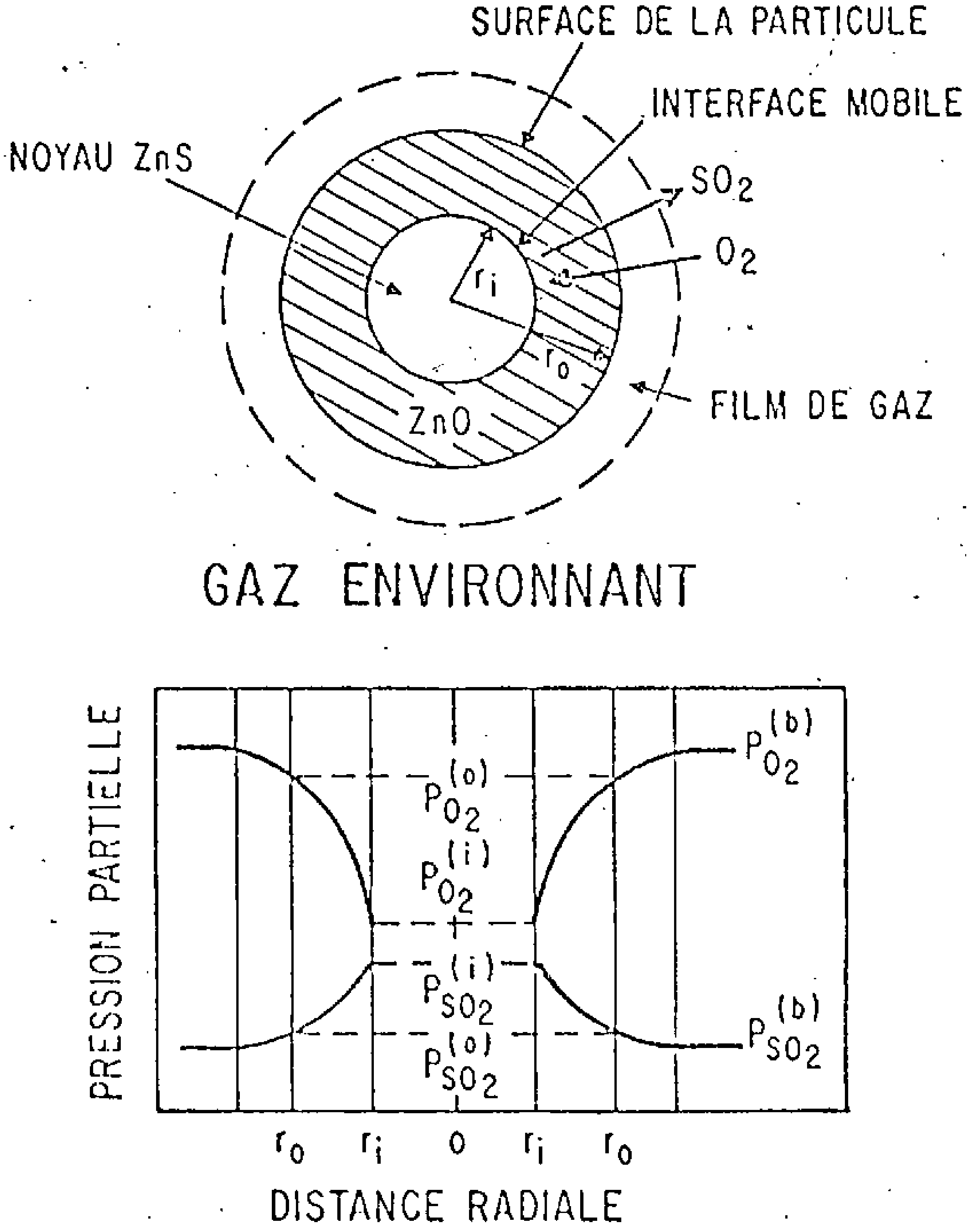
Figure IV



MODÈLE GÉNÉRALISÉ POUR L'OXYDATION D'UNE SPHÈRE DE ZnS.

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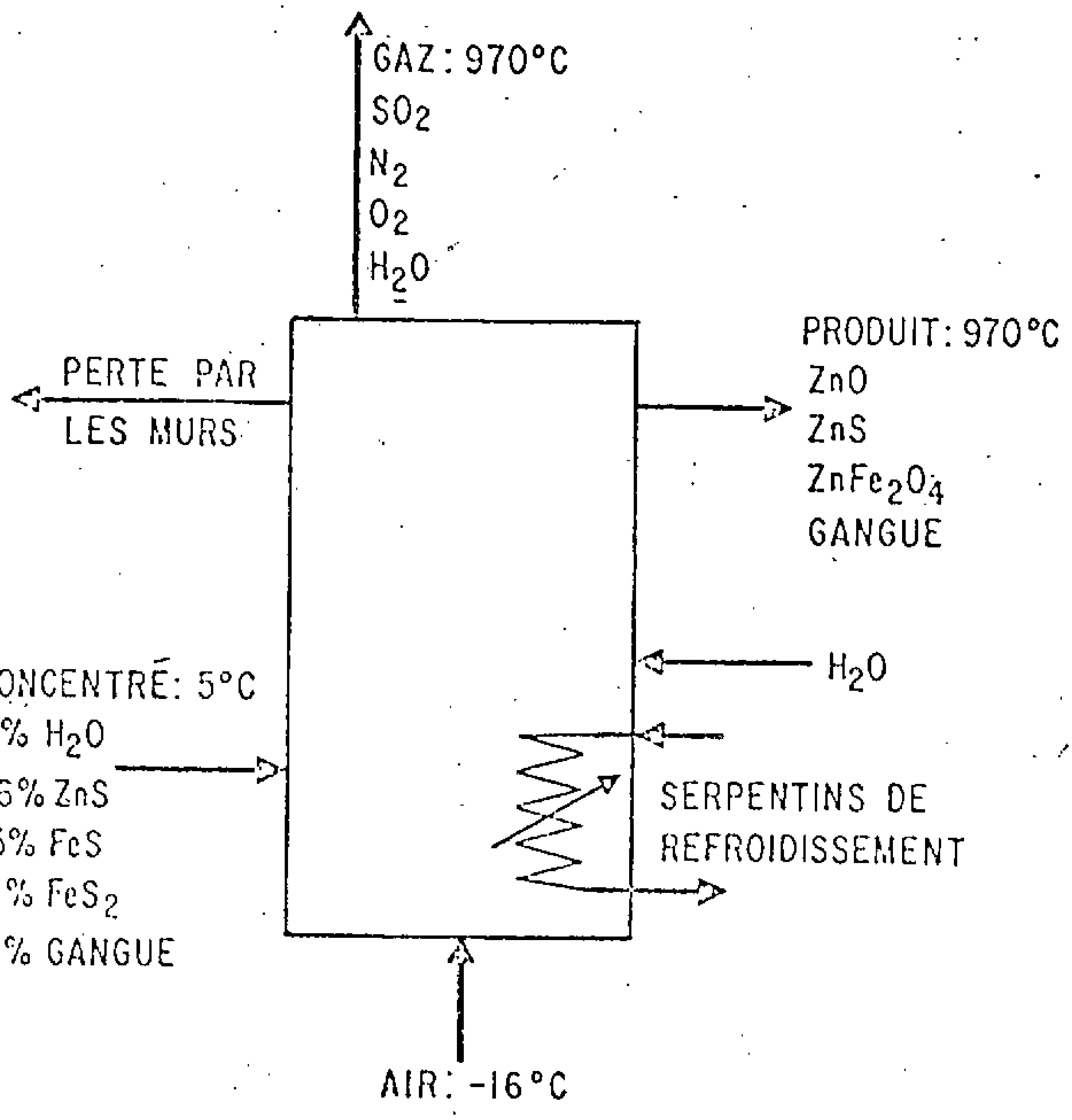
Figure V

SCHÉMA DES ENTRÉES ET SORTIES D'UN FOUR À LIT FLUIDISÉ



5) Bilan de masse et d'énergie

Le bilan de masse et d'énergie est l'outil de base pour la compréhension d'un procédé tel que le grillage. La figure V aidera à visualiser toutes les entrées et sorties du grilleur. Il est à noter que les valeurs don-

.../ 12

nées dans ce bilan ont été prises sur le four de 450 tm/j de C.E.Z. Ces valeurs peuvent varier légèrement dans le temps. Le bilan considéré a été fait pour une période de <u>une (1) heure</u> et a été simplifié pour ne pas trop alourdir la présentation. La température de référence pour les réactions est de 25°C.

a) Bilan de matière

i) Entrées

Les entrées sont composées du minerai alimenté, de l'air soufflé et de l'eau de refroidissement et de granulation ajoutée au concentré.

L'alimentation du four fut de 21,000 kg de minerai concentré (à 5°C). Ces 21,000 kg se divisent ainsi:

		% massique sec
Zns	15,338 kg	76
FeS	3,027 kg	15
FeS_2	605 kg	3
"gangue"	1,211 kg	6
H_2O	819 kg	-

L'eau ajoutée directement dans le lit ou au concentré représente 1,560 kg.

Pour fournir l'oxygène requis pour les réactions et aussi l'air nécessaire à la fluidisation, 38,760 m^3 (réf. 0°C) d'air ont été soufflés dans le four à une température de -16°C. (Le test a été fait en hiver).

Le total des entrées représente donc 72,475 kg.

ii) Sorties

Les réactions chimiques modifient la composition, mais, à la sortie, la masse doit rester constante.

La masse du minerai grillé ne représentera plus qu'environ 85% de la masse initiale. La perte de masse provient du fait que le soufre a été remplacé par de l'oxygène.

Dans le minerai grillé, les différents composés se répartissent comme suit:

		% massique sec
ZnO	11,047 kg	64
$ZnFe_2O_4$	4,758 kg	28
ZnS	192 kg	1
"gangue"	1,211 kg	7

.../ 13

La "gangue" représente le restant des composés (Cu, Cd, Pb, SiO_2, CaO...), ainsi que le $ZnSO_4$ qui s'est formé lors du refroidissement. Ceci a pour conséquence d'introduire de petites erreurs.

Le gaz à la sortie du four est à 970°C et est composé de:

		% volumique sec
H_2O	2,579 kg	-
SO_2	12,802 kg	12.3
O_2	1,678 kg	3.2
N_2	38,275 kg	84.5

La masse totale des sorties fut de 72,341 kg.

b) Bilan d'énergie

Le grillage d'un minerai de zinc est un procédé autogène. La chaleur fournie par les réactions d'oxydation est suffisante pour maintenir une température de 950-1000°C dans le four. Afin de contrôler celle-ci, on ajoute de l'eau directement dans le lit ou avec le concentré. En plus, on utilise des serpentins de refroidissement dans le lit. Ils sont normalement au nombre de 6, mais, lors de la prise des mesures il n'y en avait que 4 en opération, ce qui peut expliquer leur contribution relativement faible pour le refroidissement (17%). Normalement environ 23% de la chaleur est absorbée par les serpentins.

i) Entrées

La chaleur provient à 100% des réactions qui se produisent dans le four. La température de référence fut prise à 25°C pour toutes les réactions.

$ZnS \longrightarrow ZnO$	- 16.34	*Gcal	73%
$FeS \longrightarrow ZnFe_2O_4$	- 5.035	Gcal	22%
$Fe_2S \longrightarrow ZnFe_2O_4$	- 1.01	Gcal	5%
	22.39	Gcal	100%

*G = 10^9

ii) Sorties

La chaleur fournie par les réactions est absorbée soit par les solides qui sont chauffés de 5°C à 970°C, par les gaz, par les serpentins de refroidissement et finalement par les murs du four.

.../ 14

La chaleur absorbée se distribue ainsi:

solide	5.38 Gcal	24%
gaz	13.06 Gcal	58%
serpentins de refroidissement	3.90 Gcal	17%
murs	0.05 Gcal	1%
	22.39 Gcal	100%

Le minerai sulfuré est un combustible qui génère 1.2 t de vapeur/t de concentré. Environ 45% de la vapeur consommée dans toute l'usine provient de la génération de vapeur au grillage.

E. CALCULS DE DIMENSIONNEMENT D'UN FOUR

La capacité d'un four à griller est en relation directe de ses dimensions. Normalement on établit la capacité du four et ensuite on le dimensionne.

La première chose à déterminer est la quantité d'oxygène nécessaire pour que les réactions d'oxydation soient complètes. Ceci se calcule à partir de l'analyse du concentré. Connaissant la vitesse de fluidisation requise, on peut calculer la surface de la sole.

Le graphique 1, représentant la grosseur des particules en fonction de la vitesse de l'air à la température du grillage, montre qu'une vitesse de 0.6 m/s est nécessaire pour avoir une bonne fluidisation.

Pour oxyder 1 kg de minerai concentré à 53.3% Zn, 33.3% S, 10.8% Fe, 0.05% Cu, 0.02% Cd et 0.01% Pb, il faut 510 g O_2. Il y a environ 3% d'oxygène en excès. Le tout conduit finalement à 1,930 l* de gaz/kg de concentré.

Pour une capacité de 450 t/j, il y aura un débit de gaz réel de 45 m^3/s. La surface de sole correspondante est de 75 m^2.

F. EVALUATION ECONOMIQUE D'UN MINERAI DE ZINC CONCENTRE

Les minerais concentrés offerts sur le marché, bien que semblables, n'ont pas la même valeur intrinsèque. A chaque élément chimique est attaché un profit ou une perte. La comparaison de la somme de ces profits et pertes, pour chacun des minerais, permet de les classifier et de choisir le plus avantageux.

Ainsi par exemple le Cd conduit à un profit parce que récupéré et vendu; le Mg conduit à une perte, car, suite à sa position dans la série des forces électromotrices, il s'accumule dans l'électrolyte et en augmente la résistance ohmique. Pour l'éliminer, l'on fait des saignées d'électrolyte que l'on neutralise.

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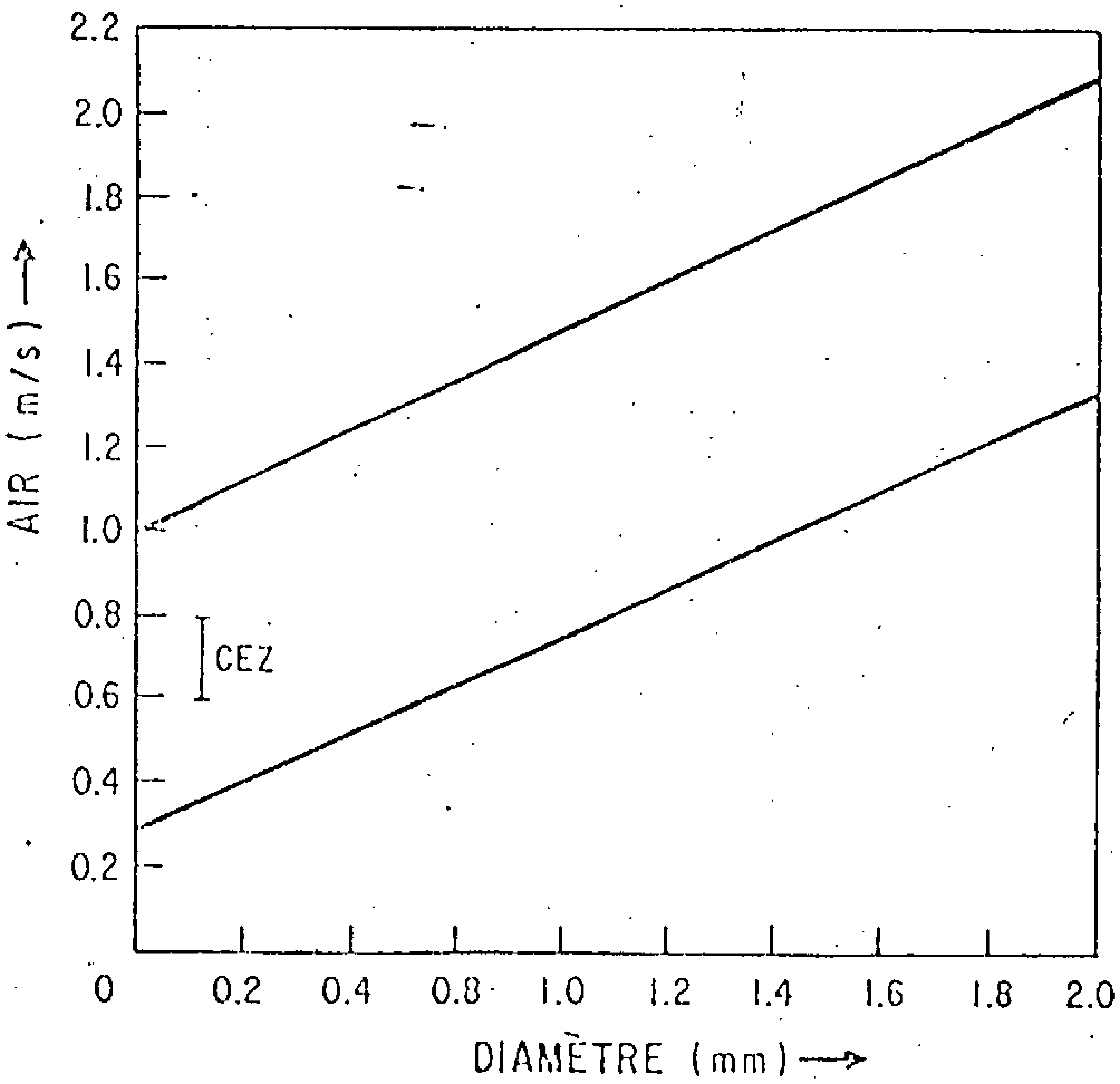
*(à 0°C et 1 atm)

Graphique I



ZONE DE FLUIDISATION INSTABLE

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Le tableau III résume la valeur qualitative des principaux éléments secondaires.

Tableau III

Valeur qualitative des éléments secondaires

Eléments	Forme de traitement	Valeur
S	Transformé en H_2SO_4	± 0
Fe	Éliminé sous forme de résidu de jarosite, de goethite ou d'hématite	-
Cd	Récupéré sous forme métallique et vendu	+
Cu	Récupéré sous forme de gâteau concentré	+
Hg	Eliminé pour maintenir la qualité de H_2SO_4	-
Cl	Eliminé lors du grillage	0
As	Eliminé lors du grillage	0
Se	Récupéré en partie après grillage	+
Co	Eliminé lors des réactions de cémentation avec Zn^o	-
Pb	Récupéré dans le résidu Ag-Pb	+
Ag	Récupéré dans le résidu Ag-Pb	+
Mg	Eliminé par neutralisation d'électrolyte	-
Mn	Eliminé par précipitation de MnO_2 et neutralisation d'électrolyte	-
Ca	Eliminé sous forme de gypse	-
Si	Dans la gangue. Poids mort éliminé avec les résidus.	-

G. PERSPECTIVES D'AVENIR POUR LE TRAITEMENT DES BLENDES

Le grillage à 1000°C, on l'a vu, a l'inconvénient de former, avec pratiquement tout le fer présent de la ferrite de zinc, $ZnFe_2O_4$. Cette ferrite est assez difficilement soluble dans l'acide sulfurique et demande des retentions et températures élevées. Le grillage sulfatant à 680°C, quant à lui, permet de contourner le problème mais a d'autres inconvénients. Les points d'interrogation que peut se poser l'ingénieur sont les suivants:

- Existe-t-il une variété allotropique de ferrite plus facilement soluble?

- Ne peut-on utiliser les propriétés magnétiques de cette ferrite pour la concentrer?

- Pourquoi n'utiliserait-on pas un autre oxydant que l'oxygène de l'air: air enrichi? oxygène en autoclave? oxydation bactérienne? acide persulfurique?

.../ 17

L'un de ces points a sa réponse:

L'oxydation directe par l'oxygène, en autoclave, devient une réalité industrielle. C'est le procédé Sheritt-Gordon dont Cominco est en train d'installer la première unité. Il a fallu vingt (20) ans pour le développer.

PD:PK:ld
21/11/80

Relatorio Mensal de Andamento ao CETEM

DIVISAO: DIMETE DATA: 2 de março de 1982

Projeto: Estagio STI/CIDA - Tecnologia de Fornos de Leito Fluidizado
Canadian Centre for Mineral and Energy Technology - CANMET
555 Booth Street, Ottawa, ONTARIO, K1A 0G1, CANADA

Relatorio no. 5

De: Jorge L.L. Brandao,
CANMET, Room 270.

Energy, Mines and Resources Canada
Science and Technology

Énergie, Mines et Ressources Canada
Science et Technologie

Your file Votre référence

Our file Notre référence

Relatorio no. 5: Fevereiro/82

No decorrer do mes de fevereiro foram realizadas as sequintes atividades:

1 - Termino dos testes com o equipamento de estudos.

2 - Continaçao dos testes com o equipmento definitivo:

- Verificaçoes do sistema de alimentaçao
- Modificaçoes na parte de saida dos gases
- Verificaçao do funcionamento do forno vertical: Adiçao de seçao de aquecimento no topo do forno vertical
- Verificaçao do pre-aquecimento e forno pre-aquecedor.
- Reparos no compressor (vacuo).

3 - Montagem do equipamento definitivo:

Após haver testado todas as partes individuais, a equipamento foi montado e testes veem sendo efetuados com vistas a determinar possiveis modificaçoes ou problemas operacionais. No relatorio de março/82, sera incluido o desenho do equipamento definitivo e detalhes das partes mais importantes.

Com a adiçao de uma seçao de aquecimento no topo do forno vertical, o gradiente longitudinal (vertical) de temperatura ficou bem proximo ao ideal com pequenas variaçoes que deverao ser atenuadas durante a ustulaçao.

4 - Visitas técnicas: (ver relatorios de visita em anexo: "Trip Reports")

i) Noranda Research
Pointe Claire, Quebec: 5 fev. 1982

ii) British Columbia Research Council (B.C. Research)
Vancouver, British Columbia: 8-9 fev. 1982

iii) University of British Columbia
Department of Chemical Engineering
Vancouver, British Columbia: 9-10 fev. 1982

iv) Cominco (Consolidated Mining & Smelting Company)
Trail, British Columbia: 11-12 fev. 1982

NOTA: Com estas quatro visitas tecnicas termina o programa de visitas previsto para esta fase do estagio.

Canada Centre for Mineral and Energy Technology,
555 Booth Street,
Ottawa, Canada.
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada,
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Canadä

Programa de atividades para o proximo mes: Março/82:

1 - Termino da montagem e modificaçoes no equipamento definitivo.

2 - Termino dos testes preliminares.

3 - Analise granulometrica do concentrado de zinco de Morro Agudo recebido em inícios de fevereiro, 1982.

4 - Inicio dos tests de ustulaçao e verificaçao do sistema de recuperaçao do produto de arraste (camara de descompressao, ciclone primario, ciclone secundario e tubo de escrubagem).

5 - Verificaçao do tempo de estabilizaçao da ustulaçao com auxilio de analises do gas efluente por cromatografia gasosa (o equipamento para tal efeito ainda nao esta em ordem).

6 - Obtençao de dados, compilaçao e analises. Lixiviaçao do produto calcinado (o equipamento para lixiviaçao ainda nao foi obtido).

7 - Elaboraçao de um programa de testes a serem coutinuados por técnicos da CANMET apos meu regresso ao CETEM (a continuacao dos testes no CANMET com o equipamento construido e montado durante a estagio vai depender da disponibilidade e interesse da seçao de metalurgia extrativa).

8 - Elaboraçao do desenho do equipamento e relatorio final.

Ottawa, 2 de março de 1982

Jorge L.L. Brandao

Jorge L.L. Brandao
eng. metalurgico

JLB/jo

Trip Report

Noranda Research, Pointe Claire, Quebec
B.C. Research, Vancouver, British Columiba
University of British Columbia, Vancouver, B.C.
Cominco - Consolidated Mining & Smelting Co., Trail, B.C.

1) Noranda Research - February 5, 1982 (Hai Teh, Phillip Mackey)
- see trip report by Linda Wilson (Appendix A)

2) B.C. Research - February 8-9, 1982 (Cedric Briens, Rod McElroy)

The purpose of this visit was to verify the existence of fluid bed reactors and projects involving fluid bed technology. B.C. Research have three fluid bed reactors used for different purposes. The first one consists of a 10 cm stainless steel tube with a fourfold expansion of the freeboard which is due to the high velocities used and short reactor tube length (see Appendix B). This reactor is being used in a project which deals with the drying and separation of coking coal from oil. Coal and oil are to be mixed and sent through pipeline from the province of Alberta to a port in British Columbia where oil and coking coal are to be shipped to Japan. There is therefore the need to develop a means by which the coal can be separated from the oil. So far tests have proven this separation to be feasible to a certain extent but an undesired percentage of oil still remains in the coal. This remaining oil can be removed by fluidizing this material at high temperatures (about 800°C) using nitrogen as fluidizing gas. The system is kept at a slightly positive pressure to avoid air infiltration. A few tests were carried out and so far it yielded good results. However, due to the nature of the material a good amount of stickiness in the overflow system as well as upstream where the oil separates from the coal, has generated some operational difficulties. At present, Cedric Briens who is the engineer responsible for this project is trying to overcome some of these problems by re-designing or modifying parts of the gas collecting system where pluggage occurs (cyclones, condenser and de-mister).

.../2.

The material is fed at the top of the reactor by a double screw feeder system which automatically adjusts its speed by a mass sensor in order to keep a constant feed (manufactured by K.-Tron, U.S.A.). No problems were reported in this area. As the material falls into the bed it dries and as it is fluidized the oil separates from the coal and is entrained as a mist, together with coal fines. The coarser coal which composes most of the feed is collected by overflow. The reactor has three overflow pipes at 12.5 cm intervals from the distributor, in order to allow variation of bed depth. The distributor is made of a perforated stainless steel plate (no. holes follows Kunii's relationship). Pressure taps and thermocouple locations can be seen on the drawing. To avoid plugging in the pressure tap, a membrane filter is used and can be blown with nitrogen from time to time in case the membrane plugs. No problems were reported, however. The most difficult part of the operation is in collecting the oil and fines. Cyclones were reported to plug often and there is still too much mist coming off the de-mister which is similar to the condenser but with wire mesh wrapped around the outlet tube inside the de-mister where the mist collects and flows down to a container.

The other two reactors are much larger in diameter and are used for coking and coal combustion. The fluid bed coker is 30 cm in diameter and its distributor was designed to allow a mixture of spouting and fluidization. This project is to be done in conjunction with Professor John Grace's staff at the University of British Columbia. The combuster fluid bed is 61 cm (24 in.) in diameter and uses a perforated steel plate as distributor. It is not being used at present.

3). <u>University of British Columbia</u> - February 9-10, 1982 (Professor John Grace)

The purpose of this visit was to discuss a few questions related to fluid bed design and the mathematical modelling of fluid bed reactors as well as to become acquainted with Dr. Grace's experiments with fluid bed reactors.

After discussing the questions I had on fluid bed design, Dr. Grace showed some of his own reference material that he thought would be helpful. These are listed at the end of this report, together with a list of articles published by the staff of his department, including his own (see Appendix C).

.../3.

Dr. Grace was very responsive to the idea of making a computer model from actual industrial operations. So far mathematical modelling has been done from a more theoretical approach and usually missing in practicallity. He asked me to keep him informed with regards to the collection of data from industry and that he would be willing to become involved in the actual modelling. I let him know that all data collected from industry would be sent to me personally and that with the approval of the companies concerned, I would most willingly forward to him any information he may require.

After the discussion period we went on a tour of his laboratories where experiments with fluid bed reactors take place. His most recent experiment is related to strains and stresses in fluid bed reactors and showed the equipment he is using which consists of a rectangular reactor, where tubes can be inserted parallel to the distributor and at various distances from it and the stresses in the tubes can be monitored by strain gauges. No prior work has been done in this area and the results shall be of direct interest to industry.

His section of the Department of Chemical Engineering has a large number of fluid bed reactors, many of which made of acrylic or Pyrex for studies at room temperature only. Some students were observing the fluidization of granulated single-size particles and the effect of velocity on bed height. Most of the work done at ambient temperature is basic to under-graduate students. At present all the work being carried out at high temperatures are related to coal combustion and gasification, and fluid bed coking in conjunction with B.C. Research.

Dr. Grace suggested that I keep in contact with the Engineering Foundation, in the U.S.A., in order to be informed of conferences in fluid bed technology around the world. The address and telex number are listed with the references at the end of this report.

4) Cominco - February 11-12, 1982 (Jim Broster, Ron Anderson)

Of all the visits I made this was by far the most interesting and a good deal of valuable information obtained.

.../4.

After briefing with Ron Hall (Assistant to General Manager, Trail Operations) and Jim Broster (Roaster-Acid plant superintendent), Jim conducted me to the zinc plant office for a meeting with top personnel of the Roaster-Acid plant:

Jim Broster:	superintendent - R/A plant
Don McKay:	Operating superintendent - R/A plant
Roy Melenka:	Assistant operating superintendent - R/A plant
Martin Meyer:	Assistant foreman - R/A plant
Bob Ross:	Technician - R/A plant
Ron Anderson:	Project engineer
Bruno Piconi:	Development engineer
Gerry Toop:	Supervisor, Technical Development Centre

During the meeting operational and technical problems were discussed and I had the oportunity to ask many questions related to their operation and also explained to them the purpose of my visit, my programme at CANMET through CIDA, and gave them a copy of the Data Collection Questionnaire. They seemed very interested in seeing the modelling of industrial operations carried out and expressed their desire to participate in this effort and were looking forward to obtaining results in the near future.

After the meeting I was taken on a tour of the Roaster-Acid plant, guided by Ron Anderson and was able to observe some of the tests they are doing on their feeding system which will be discussed later in this report. After having been shown the entire roasting and acid plants we broke for lunch with some of the R/A plant staff and Bill Jewitt, Manager of Zinc Operations. During this informal gathering I explained once more the purpose of my visit and stressed the part about mathematical modelling, and, once more, felt good reaction on their part.

In the afternoon I was asked to sign an agreement of non-disclosure of details of what I was about to visit which were their new pressure-leach (Sherritt Gordon-Cominco process) plant and their brand-new Electrowinning and Melting (E&M) plant where I had the opportunity to see what seems to be the most up-to-date technology in what will be a highly automated plant. The E&M plant is only ½ complete and operating only at 1/6 of its capacity. I saw their automatic stripper, melting furnaces, their very large piling and loading area, and the section of the melting plant where "dross" is made in a fluid bed grinder and sent to the zinc plant where the leach liquor is purified before it is sent to the E&M plant. One of the greatest advantages of the new E&M plant, for the operational point of view, is that they are both under one roof, unlike their old set-up. From the engineering point of view there is excellent control of the process variables which will make possible greater production at high efficiency and quality of product. There seemed to be extensive use of radioactive level controls with minor problems reported. The whole new set up is a very impressive site. The other side of the plant, their Lead Plant, which I did not have time to visit will also undergo a complete technological change when their conventional process is replaced by the Russian Kivcet Process. It is almost breath-taking to witness such an enormous company, like Cominco at Trail, transforming itself and embracing state-of-the-art technology, obviously not without reluctance, but nonetheless with great courage and pioneering spirit. It will be very interesting indeed to observe how the new plants behave both technologically as well as economically within the next decade or so.

Cominco's Fluid Bed Roasters:

The roaster plant has two standard Lurgi roasters, twins, of 84 m^2 distributor area and 8000 tuyeres per distributor. Each reactor has its boiler-settler, twin primary-cyclones in parallel, and a "Wheelabrator" electrostatic precipitator operating at about 400°C with 80-90% efficiency, and a scrubber circuit before entering the acid plant.

The reactors have an overflow port about 1 m from the distributor plate, where the coarse product flows continuously and accounting for 30-35% of the total product. Sixty-five to 70% of the product is collected in the gas cleaning system being 30-35% in the boilers, 30-35% in both cyclones, about 1% in the ESP and less than 1% in the scrubber. Both reactors operate between 875°C and 975°C depending on the amount of Pb and Fe in the feed. The more Pb and Fe the lower the temperature in order to avoid "freezing" of the bed caused by the formation of an eutectic at higher temperature. A large percentage of the iron in the feed forms a ferrite with the zinc during roasting. This is due to the fact that most of the iron is already in the sphalerite structure replacing some of the zinc in what is called a "ferroan sphalerite" or Marmatite ((Zn,Fe)S, where 6:5 > Zn:Fe > 5:1). As the reaction between zinc and iron

.../6.

with oxygen takes place the proximity of both Zn and Fe atoms is such that they combine to form zinc ferrites instead of separate oxides.

Sulphide/Sulphur ratio in entrained calcine is 0.5-0.9 whereas in the overflow it is about 0.1. The superficial gas velocity is estimated to be between 30-60 cm/sec but no one was certain of particle size of distribution. One of the problems they are trying to solve is homogeneity of moisture in the feed which they believe will contribute to increase retention time without decreasing production. On each reactor they have insta led a different feed mixer. One of them has Lurgi's standard "P-K" mixer and the other an "Eirich" mixer-granulator.

As the feed receives water sprays before falling onto the slingers there is a larger % moisture at the top (about 10%) whereas the bottom part remains the same (about 4%). This means that the dryer material will spend less time in the bed than the moist agglomerated material. By mixing well the feed with the moisture more agglomerates can be formed and therefore carryovers or "short circuits" may be lessened. So-far tests have hinted that the "Eirich" mixer improves reactor performance. The only disadvantage is that many foreign bodies (such as a small aluminum washer) can jam the mixer and even bend the shaft in its somewhat delicate mixing mechanism. Good screening is therefore a must as well as a back-up system in order to avoid interruptions of the feed.

Since both reactors seem to be run already above their nominal capacity, the entire gas cleaning system becomes overloaded and problems such a build-ups in boiler bundle-tubes and coils are frequent. Due to the design of their plant there is no space available for adding extra tube-bundles to the boilers or expanding them. Perhaps an extra cyclone could be added but would not lower the load off the boilers. In designing a plant one should always leave a good tolerance for future expansion.

In comparing Cominco's roasting operation with that of CEZ's I found interesting to verify that whereas at CEZ a 98% efficiency in their ESP is considered low, at Cominco it did not seem a great concern to have efficiencies of the order of 80-90%. Cominco say they recover all remaining calcine in the scrubber so that it is not a great problem to have such ESP efficiencies, although CEZ also have a scrubbing circuit. If both CEZ and Cominco decide to fill out the Data Collection Questionnaire one is bound to find other discrepancies as well as reasons for their existence.

.../7.

Apart from the twin Lurgi reactors Cominco's roaster plant has what is called a suspension roaster which was developed from an old multiple-hearth furnace where the middle hearths were removed. The material is fed from the top and free-falls to the bottom where it is collected as calcine. Its energy efficiency is much lower than that of a fluid bed reactor and, also, it does not allow interruptions in the feed which are longer than 10-15 seconds, before losing temperature and having to be re-heated by burners to attain reaction temperature once feeding is resumed. The Lurgi reactors can stand up to 60 minutes (1 hour) feed interruption with no required re-heating. Also, from an industrial hygiene point of view the suspension roaster creates dust build ups all around it. This dust, along with all dusts which accumulate in the plant is being vacuumed and recycled to the feed system. This practice has been so successful it is now being expanded.

A basic technical description of the main production facilities can be found in Appendix D.

Jorge L.L. Brandao
Metallurgical Engineer

APPENDIX A:

- Trip Report: Noranda Research,
Pointe Claire
February 5, 1982

by Linda Wilson

Energy, Mines and Resources Canada Énergie, Mines et Ressources Canada

CANMET

MINERAL SCIENCES LABORATORIES
INTERNAL REPORT

VISIT TO NORANDA RESEARCH CENTRE, POINTE CLAIRE, QUEBEC; FEBRUARY 5, 1982

L.J. Wilson
Metallurgical Chemistry Section
Extractive Metallurgy Laboratory

February 1982

Project: MRP-3.5.0.0.05
Control of Toxic Pyrometallurgical Emissions

Field Trip ☒ Plant Visit ☐ Conference, Convention, Symposium ☐

Test Report ☐ Translation ☐

Contains our notes ET

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VISIT TO NORANDA RESEARCH CENTRE,
POINTE CLAIRE, QUEBEC;
FEBRUARY 5, 1982

by

L.J. Wilson

Personnel Contacted

Phillip J. Mackey:	Program Manager - Noranda Process, Noranda Mines Limited
C. Hai Teh:	Department of Chemical Engineering, Noranda Research Centre

Discussions were held with C. Hai Teh on the following topics.

1. Fluid Bed Roaster

A laboratory scale fluid bed roaster (see Figure 1) has been constructed to investigate the sulfation roasting and dead roasting of metal concentrates. At present, the roasting of copper concentrate during a single pass through the reactor is being studied.

The roaster consists of a 4" I.D. stainless steel pipe 4 feet in height (straight freeboard). The bed height can be varied since there are four discharges, at one foot intervals. The roaster is wrapped in Kanthal wire and heated electrically. The stainless steel distributor plate has twenty-one holes each 1/3" in diameter. The pressure below the distributor plate is 1 psig (about 700 mm H_2O).

The dry concentrate (<0.5% H_2O, -100 mesh) is fed via a screw feeder (3/8" I.D.) at 1½ kg/h to the roaster. The screw feeder has been carefully constructed so that there is very little dusting. During operation it has to be air-cooled to prevent sintering of the concentrate in the screw.

The bottom of the roaster is run at 600-800°C and the top at 800°C. At these temperatures and the operating velocity of 30-50 cm/s a concentrate feed or a calcine bed will sinter. To prevent this, the fluid bed includes 1 foot of sand (200-300 microns). Retention time in the fluid bed is 30-4 minutes.

Entrained calcine (30% Cu, 10-20% S) is collected in the two hot cyclones. The cyclones are heated to prevent the condensation of acid and volatiles (arsenic). An electrostatic precipitator follows the cyclones in the collection train. It actually functions as a settling chamber because of power supply problems. Sixty-80% of the calcine is collected in the two cyclones while 10% is collected in the settling chamber. A sodium hydroxide spray scrubber removes the volatiles from the gas stream which then passes through a wire mesh demistor and is vented.

A gas sampling point is located between the settling chamber and the spray scrubber. Oxygen is metered continuously while other gases are analysed periodically on a gas chromatograph. The system is run under a positive pressure so that the gas analyses will not be affected by leaks. Stainless steel Swagelok fittings have been used for all connections and have been successful in preventing leaks.

The apparatus requires a 3-hour stabilization period prior to the 3-hour test period. Many variables (temperature, gas flow, feed rate) are changed from run to run. The results for a repeated test are 80-90% reproducible.

The system is too small to extract chemical and metallurgical scale-up data. If the tests are successful, a pilot plant will be constructed to obtain this information.

2. Arsenic Distribution

In 1974 an arsenic balance was done on the continuous smelter at Noranda, Quebec to determine the arsenic in the work environment from an occupational health viewpoint. Since then, the arsenic distribution has not been studied. No arsenic balance has been done on the laboratory scale fluid bed roaster.

Arsenic as As_2O_5 dust is removed from the gas stream by the hot electrostatic precipitators while arsenic as volatile As_2O_3 passes through them. Presently, Noranda is investigating possible methods of collecting volatile arsenic. They have taken two approaches: (1) to react the arsenic with a substance to yield a solid product which will collect in the Cottrells and (2) to pass the gas through a packed or fluid bed of reactants which will fix the arsenic and thus remove it. The tests are done initially with a gas stream of nitrogen and, if successful, are repeated with a simulated smelter gas.

3. General Tour

Three research pilot plants were observed. In the Physical Metallurgy Pilot Plant activities are centred on metal casting and alloy development. Alloy activities are related principally to zinc.

Activities in the Chemical Engineering Pilot Plant encompass chemical processing and high temperature metal extraction and refining. Some of the equipment available includes a pilot converter, a fluidized bed roaster, a plasma reactor, and a barrel furnace for smelting lead. Some work has been done on the separation of solids from process gases and a specially designed flue system has been developed.

The Electrochemistry Pilot Plant is concerned with the final steps of metal recovery such as zinc electrowinning processes, and electrochemical treatment of dilute or waste solutions in order to recover metal values or regenerate reactants. Investigations on novel battery systems are also being done.

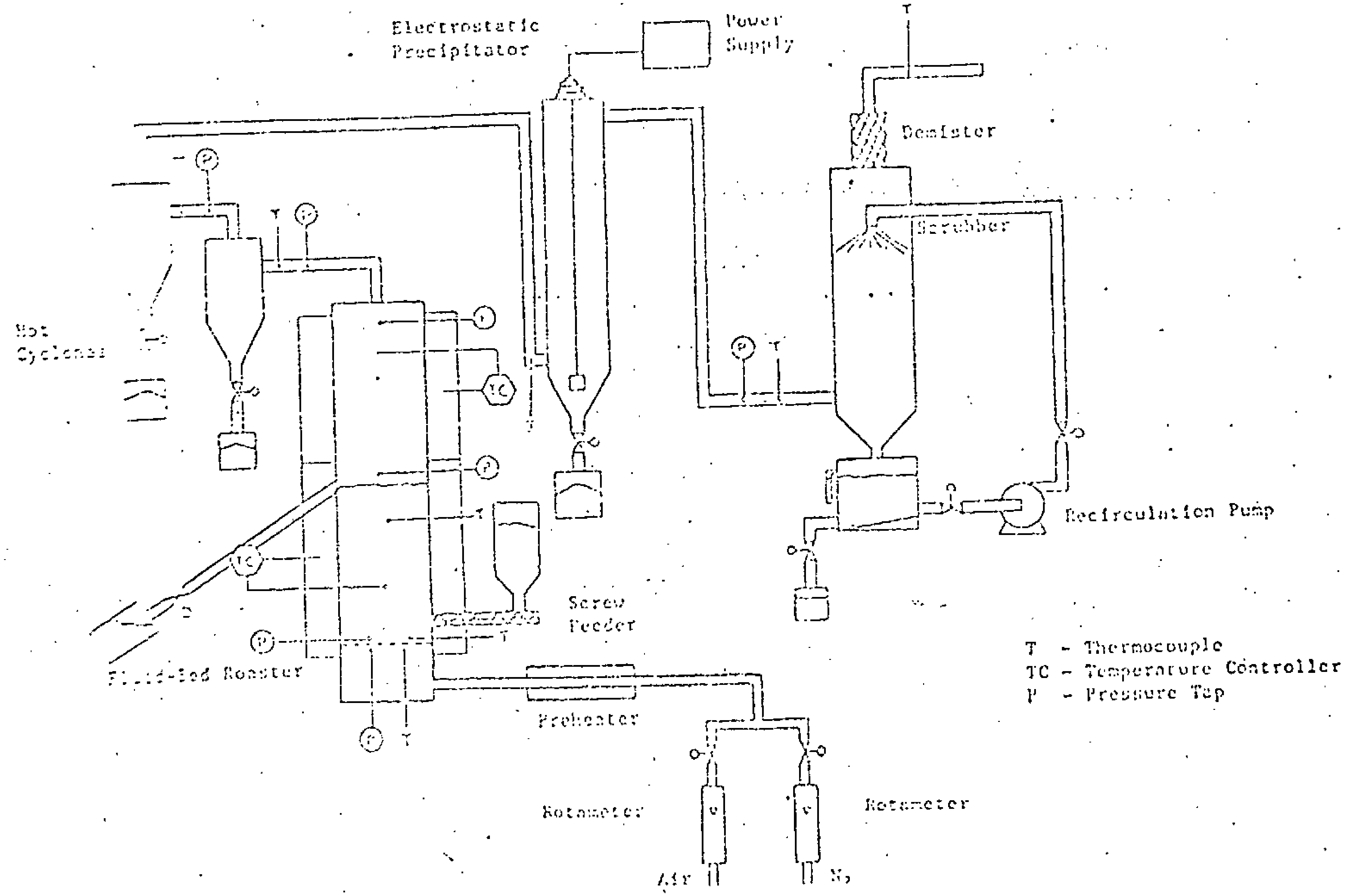
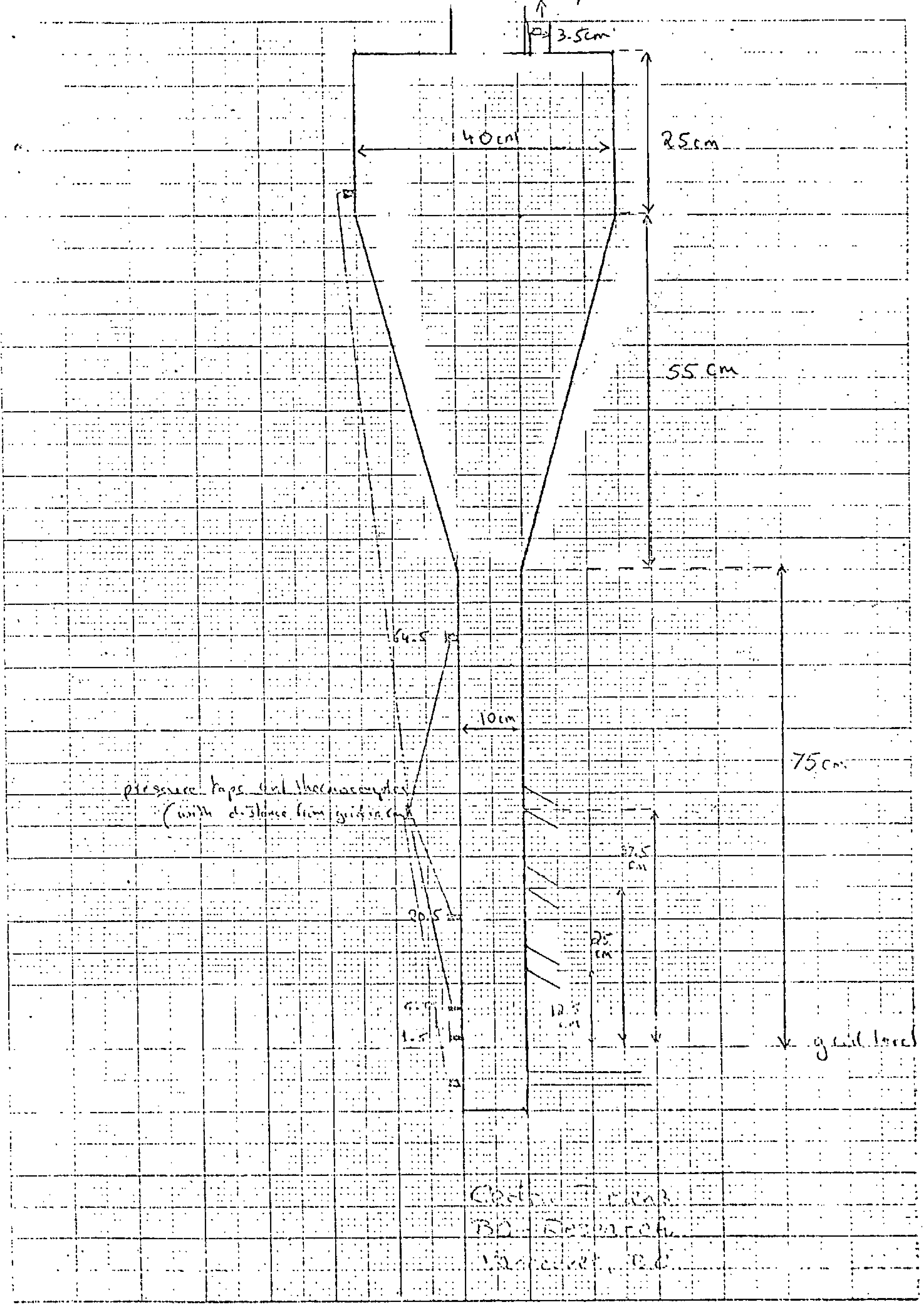


Figure 1 - Schematic Diagram of the Laboratory Fluid-Bed Roaster and its Ancillary Equipment.

CONFIDENTIAL

APPENDIX B:

- Drawing of small pilot fluid bed reactor at B.C. Research, by Cedric Briens


3.5cm
40cm
25cm
55 cm
64.5
10cm
75cm
pressure taps and thermocouples
(with distance from grid in cm)
37.5 cm
20.5
25 cm
6.5
1.5
12.5 cm
grid level
[illegible]
BD Research
[illegible]

APPENDIX C:

- Department of Chemical Engineering, U.B.C.
- List of active research projects
- Recent Publications
- Dr. John Grace reference material

THE UNIVERSITY OF BRITISH COLUMBIA

DEPARTMENT OF CHEMICAL ENGINEERING

LIST OF ACTIVE RESEARCH PROJECTS

(as of January 1982)

Branion, R.M.R.	Microbial leaching of zinc sulfide concentrates Drainage on paper machines Mechanism of anaerobic methane formation Fluid mechanical behaviour of jets in papermaking
Cavers, S.D.	Axial mixing in liquid-liquid extraction spray columns Design procedures for non-mechanically agitated liquid-liquid contactors
Epstein, N.	Colloidal particle deposition and release Pulsed three-phase fluidization Modeling ZnS_2O_4 synthesis in 3-phase fluidized beds Comparison of two heat transfer fouling probes Sedimentation potentials in hindered settling Spouted bed combustion Spouted bed and spout-fluid bed mechanics
Grace, J.R.	Interphase mass transfer in fluidized beds Forces on tubes immersed in gas fluidized beds Coal combustion and pyrolysis Solar energy storage Mechanics of spout-fluid beds Flow regimes in gas-solid flows High pressure fluidization
Kerekes, R.J.	Pulp flocculation in process flows Mixing in pulp suspensions Sprayability of concentrated black liquor
Lielmezs, J.	Fluid flow in freezing porous media Studies in transport properties Magnetic field effect on thermodynamic and transport properties of liquids Magnetic field effect on chemical reaction rates Thermodynamic and transport properties of pharmaceutical fluids Studies in thermodynamics Equations of state

eisen, A.	Production of slow release fertilizers Analysis and prediction of ambient air data in terms of frequency distributions Degradation of amine treating solutions. Fluidized bed Claus reactor studies Supercritical fluid extraction of hydrocarbons Oxidation of Methane to Methanol
urray, F.E.	Catalysis in the oxidation of sulphide in aqueous solution
loman, C.W.	Design of electrochemical reactors Electrosynthesis of peroxide Electrosynthesis of sodium dithionite Electrosynthesis of quinones Electrokinetic studies of oxygen reduction Air depolarised electrodes in chlo-alkali industry Removal of chloride from pulp mill black liquor
inder, K.L.	Solar heat storage Catalytic direct reduction of Na_2CO_3 to Na_2O Rate of crystallization of Na_2SO_4 from acid solutions Compressive behaviour of skin components Bacterial masking in anaerobic fermentation Direct contact evaporative heat transfer Modelling of blood to lymph transport Non-Newtonian flow through porous media Adaptive control systems
Thompson, D.W.	Laser-holographic visualization of the wall pressure field in turbulent flow Fouling in membrane separation processes Algorithms for computation of multicomponent, multiphase equilibria Modelling of distributed contacting and chemical reaction CHEMOS--A chemical engineering modelling and optimization system
Watkinson, A.P.	Anodic oxidation of coal processing effluents Gasification of coal and wood in spouted and fluidized beds Coal pyrolysis and combustion in spouted beds Particle dynamics, heat flow and calcination in rotary kilns Scaling of heat transfer surfaces Comparison of heat transfer fouling probes

RECENT PUBLICATIONS - Dept. of Chemical Engineering - U.B.C.

DR. R.M.R. BRANION

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2. Branion R.M.R., Hauptmann E.G. "The Current State of Drainage Theory" accepted 1979 for publication as a monograph by the Tech. Sect. of Can Pulp and Paper Assoc. approx 130 pages. Portions were presented at the 1977 meeting of the CPPA.
3. Mardon J., Branion R.M.R. "Drainage at the Wet End" 1977 *Tappi* 60(7), 12-13 - a technical editorial requested by the editor of *Tappi*.

DR. STUART D. CAVERS

1. Lim, C.J., J.E. Henton, G. Bergeron, and S.D. Cavers, 1977. "Internal Sampling of Liquid-Liquid Spray Columns at Steady State. II. Application to Mass Transfer Experiments". Proceedings of the International Solvent Extraction Conference, ISEC 77 (Toronto), Vol. 1, 248-255, Can. Inst. of Min. & Met., Montreal (1979).

2., 3., 4. One paper each year on how, where, and at what salary the members of our graduating B.A.Sc. class are employed after leaving U.B.C. The latest of this series (published in *Chemistry in Canada*) is:

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5. Paper presented - Horvath, M., and S.D. Cavers, 1979. "The Effect of Vertical Alignment on Continuous-Phase Axial Mixing in a Pilot Liquid/Liquid Spray Column", 87th National Meeting, A.I.Ch.E., Boston, Mass., Aug. 19-22, 1979.

DR. NORMAN EPSTEIN

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September 1976

4 - Fluid Bed Technology
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NOTE: This book is to be released sometime in 1983

5 - Engineering Foundation Conferences
345 East 47th Street
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Telex no. 126022

ROASTING ZINC CONCENTRATE IN
FLUIDIZED BEDS - PRACTICE AND PRINCIPLES

By

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ABSTRACT

Detailed design and operating data are given for a 230 tonne/day, 6.4 m i.d. zinc concentrate roaster at Canadian Electrolytic Zinc Limited, Valleyfield, Quebec. The operating data are interpreted with respect to some important fundamental concepts in fluidization such as the minimum fluidizing velocity, the two-phase theory, the reactor models based on bubble mechanics, elutriation rate constant and solids residence time distribution. Important areas that require further research are identified with the emphasis on fluidization.

INTRODUCTION

Davidson and Harrison(1) wrote that "..... fluidization has acquired one of the characteristics of Shakespearean criticism: the volume of literature is such that it cannot be read in a lifetime." Most of this literature originates from laboratory research and deals with the science and technology of fundamental concepts. And yet, the design of industrial fluidized beds remains largely an art. The fluidized bed roasting of zinc concentrate is one such example. Heath(2) describes the world's largest zinc roaster at the Onahama plant of the Toho Zinc Company. The 13.4 m diam Dorr-Oliver reactor has a capacity of 800 tonnes/day. The distributor contains more than 2000 individual tuyeres and supports a fluidized bed weighing about 400 tonnes. It seems appropriate to try and bridge the gap between the science and technology of fluidization and the industrial operation of large beds. This could serve as feedback to researchers in their quest for large scale data.

The three objectives of this paper are:

1. to provide detailed design and operating data on an industrial fluidized bed zinc roaster at Canadian Electrolytic Zinc Limited in Valleyfield, Quebec.

2. to qualitatively relate some important concepts in fluidization to the industrial operation of the roaster; in particular, the minimum fluidizing velocity, the two-phase theory and the bubbling bed reactor models, elutriation and particle residence time are discussed.

3. to identify some areas of research that may profitably be undertaken at Universities that may help in the design of larger, more efficient reactors with higher capacities and better calcine quality.

The emphasis throughout this paper is on fluidization. Important points such as, the sulphide and sulphate content of the calcine, calcine cooling, dust recovery from the waste heat boiler, cyclones and electrostatic precipitator, minor element distribution, agglomeration and build-up on the bed coils, which are of interest to the operator are not discussed.

The roasting of zinc concentrate is characterized by a heterogeneous exothermic oxidation which may be considered irreversible. The primary reaction is

$$ZnS(s) + \frac{3}{2} O_2(g) \rightarrow ZnO(s) + SO_2(g) \quad \ldots\ldots\ldots\ldots\ 1.$$

The reaction is carried out in refractory lined vessels with no auxiliary heating (except for start-up); and usually cooling is necessary to maintain the bed temperature between 950 to 1000°C.

Two identical roasters came on stream at Canadian Electrolytic Zinc Limited at Valleyfield Quebec in late 1965. Valleyfield is on the St. Lawrence River about 55 km south-west of Montreal.

ZINC [illegible] AT CANADIAN ELECTROLYTIC ZINC LIMITED

There are two parallel roasting trains. Each train consists of a feed bin, a Lurgi Turbulent Layer roaster, a forced circulation waste heat boiler, two dust cyclones and a Lurgi two-field electrostatic precipitator. Heino et al(*) give a general description of the roasting facilities including the Monsanto contact sulphuric acid plant and the Ugine-Kuhlman phosphate fertilizer plant. Zinc concentrates are received from three different mines in Northern Ontario and Quebec. The material is unloaded from 65 tonne flat-bottom railway cars to a 2500 tonne storage bay in the unloading shed. The following is a description of one of the two similar roasting trains.

The Roaster Plant

The process flowsheet of the roaster plant is shown in Figure 1. Zinc concentrate is conveyed from a 250 tonne storage bin to an 80 tonne feed storage bin. It is then discharged from the feed bin onto a variable speed conveyor belt which delivers the blende to a weight belt. From the weight belt, the material is sent to a high speed slinger belt feeder which slings the concentrate into the reactor through an open side-port. The moisture content of the concentrate is typically 5.3%. In addition, approximately $189 \times 10^{-6} m^3/s$ of water is sprinkled over the concentrate on the feeder belt. This added water tends to agglomerate the fine particles for better feeding and helps to control the temperature of the bed and freeboard.

A 187 kW centrifugal blower supplies combustion air to the conical windbox of the fluidized bed. The off-gas, containing about 75% of the total calcine product as entrained dust, leaves the expanded freeboard through a side-port at the top and passes into the waste heat boiler.

The waste heat boiler is forced circulation with an external steam drum. The hot dusty gas at about 990°C enters a water-cooled settling chamber before passing through four bundles of boiler tubes which are suspended in the gas stream. The gas leaves the boiler at about 320°C. The fluidised bed also contains cooling coils to help control the mean bed temperature. These bed coils are an integral part of the waste heat recovery system. Water at 250°C is pumped from the steam drum and forced to circulate in parallel through the bed coils and the boiler coils in a closed circuit. Process steam is withdrawn from the steam drum. The total production rate of steam from the boiler and bed coils is approximately 11.5 tonnes/hr at 250°C and 39.1 atm. The specific steam rate is about 1.2 tonnes steam/tonne of concentrate fed, of which the bed coils contribute about 10%. Each tube bundle in the boiler is equipped with a mechanical rapper which removes accumulated dust. In addition, the boiler is lanced with high pressure air once each shift to recover the dust. The dust falls into hoppers at the bottom of the boiler and this calcine is subsequently discharged onto a drag conveyor. Approximately 60% of the total calcine is recovered from the waste heat boiler. The pressure drop across the boiler is typically 0.2 mm Hg.

The gas from the waste heat boiler is split into two parallel streams. Each stream passes through a cyclone 2.5 m in diameter at the top and 8.2 m high. Approximately 12.5% of the total calcine is recovered from these two cyclones. The dust is discharged onto the calcine conveyor belt. The gas leaves the cyclones at about 315°C and the pressure drop is typically 4.3 mm Hg.

The gas streams leaving the cyclones are reunited before entering the Lurgi two-field electrostatic precipitator. The precipitator consists of frames of collecting plate electrodes and discharge wire electrodes. The dust loading in the gas is reduced from 9100 mg/Nm³ to about 120 mg/Nm³ so that the precipitator recovers approximately 2.5% of the total calcine. The temperature of the gas leaving the precipitator is 310°C and the pressure drop is about 2 3 mm Hg so that the draft at the outlet is -7.1 mm Hg.

The cumulative calcine product from the bed overflow, the boiler, the cyclones and the electrostatic precipitator is conveyed to storage. Subsequently, the calcine is leached and the zinc electrowon. The SO_2-rich gas leaving the electrostatic precipitator is sent to the Monsanto contact plant for the production of sulphuric acid. Some of the sulphuric acid is used for the production of fertilizer in the Ugine-Kuhlman phosphate plant and the remaining acid is sold.

The Fluidized Bed Roaster

The cross-section of the Lurgi Turbulent Layer roaster is shown in Figure 2. The outer steel shell is 19 mm thick. It is lined with 150 mm of insulating brick and 230 mm of refractory brick. The inside diameter of the lower hearth is 6.28 m which extends for 3.58 m above the distributor. Then the inside diameter increases over a height of 4.01 m to the expanded freeboard diameter of 9.68 m. The gas outlet is approximately 2.4 m square and its centerline is 10.20 m above the distributor. The distance from the distributor to the cord of the dished roof is 12.03 m. The dished roof has a radius of 9.14 m and contains a safety explosion flap plug valve.

Concentrate is horizontally slung into the bed through a 250 mm diam opening at a level 1.7 m above the distributor. Product calcine from the bed is collected through an overflow discharge chute. There are also underflow discharge pipes to empty the bed which are seldom used. The conical windbox contains a discharge port to remove bed material which dumps through the distributor. The material which accumulates in the windbox is removed twice per shift.

The distributor contains approximately 3300 individual tuyeres set in castable refractory. The design and configuration of the tuyeres are shown in Figure [illegible]. The tuyeres are cast steel tubes, 186 mm long, 35 mm o.d. and 22 mm i.d. tapered inside to a 6 mm diam orifice. Each tuyere is tack welded to a steel support plate and refractory is cast between the tuyeres to the top of their surface. The tuyeres are arranged in a 102 mm square array.

The reactor is supplied with three bundles of coils positioned at 120° intervals. The coils extend horizontally into the fluidized bed. These bed coils help to control the mean bed temperature. The oil burners, shown in Figure 2, are used to preheat the fluidized bed during cold starts. When the mean bed temperature has reached 700°C, concentrate is fed to the bed and the reactor quickly reaches its final operating temperature of about 990°C.

Operating Data

The operating conditions of the roaster vary and depend upon the quantity, composition and particle size distribution of the feed. For instance, Heath(2) has reported that excessive lead and copper in the concentrate may promote agglomeration in the bed which will lead to localized de-fluidization. He also states that these metals tend to increase the total sulphur in the calcine. In addition, the amount of excess air used for roasting affects both the sulphide and sulphate content of the calcine. It is desirable to minimize the sulphide sulphur since this represents a direct loss of zinc. On the other hand, some residual sulphate may be required to produce make-up acid in the subsequent electrolytic plant. However, in some plants, sulphate accumulation is a problem. The mean bed temperature also affects the quality of the calcine. In the present case, operating above 950°C has been found to produce a calcine with low sulphide sulphur with acceptable sulphate sulphur. For illustrative purposes, the operating conditions of one of the two roasters at Canadian Electrolytic Zinc have been averaged over a period of one day in July 1974. Pressures, temperatures and flowrates are recorded on an hourly basis in a daily log sheet. These recordings are then collated into daily and monthly summary reports.

The roaster is fed with three different concentrates. The typical weighted average analysis is given in Table I. Approximately 80% of the concentrate is ZnS; the rest being mainly sulphides of iron and copper. The flowrates of solid and gas through the roaster were averaged over one day and are recorded in Table II. The concentrate feedrate and total calcine produced are measured quantities. However, the product distribution between the bed overflow, boiler, cyclones and precipitator were calculated by material balance and confirmed by estimates based on some measurements. The air flowrate to the roaster is measured using an orifice meter. The concentration of SO_2 in the gas is monitored after the electrostatic precipitator. The recorded feedrate of concentrate in Table II includes the 5.8% moisture but does not include the water addition before injection into the bed.

The pressure, temperature and dust loading in the gas stream are summarized in Table III. The pressure drop across the distributor, 60 mm Hg, was estimated from measurements on a single tuyere.

Grab samples of concentrate, bed overflow, cyclone product, precipitator product and total calcine are taken on a regular basis. At the end of the month, the samples from each stream are mixed and sieved to give a composite particle size distribution for each solid stream. The results for the month of July 1974 appear in Table IV. The boiler product and elutriated solids particle size distribution were calculated from a material balance.

The operating data in Tables I to IV will be used to demonstrate the applicability of some fundamental concepts which are considered basic in fluidization.

FLUIDIZATION ASPECTS OF THE ROASTER

Minimum Fluidizing Velocity

Calculation of the minimum fluidizing velocity is usually a good starting point in defining the type of fluidization in the roaster. Kunii and Levenspiel(4) present a correlation derived from 284 data points with a standard deviation of ± 34%. The minimum fluidizing velocity Uo is given by

$$U_o = \frac{\bar{d}^2 (\rho s - \rho g)\, g}{1650\mu} \; \frac{cm}{s} \text{ for } Re_o < 20 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2.$$

They suggest that the surface area mean particle size $\bar{d}$, defined by

$$\bar{d} = \left(\sum_{all\, i} \frac{p_i(d)\Delta d_i}{d_i} \right)^{-1} \; cm \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.$$

is the most suitable to use in equation 2. Equation 3 states that the external surface area of a distribution of sizes is the same as that for a mono-size of diameter $\bar{d}$ for equal weights.

Another important correlation based on a wide range of data is given by Leva(5)

$$U_o = \left(Re_o^{-0.063} \right) \left(\frac{\bar{d}^2 (\rho s - \rho g)}{1430\mu} \right) \frac{cm}{s} \text{ for } Re_o < 5 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 4.$$

Leva also determined that equation 3 is the best for correlating beds of wide particle size distribution. This is not surprising since particle surface area is the important parameter for pressure drop in flow through particulate beds.

For the present case, the application of equations 2 and 4, to the experiment in Figure 4. However, equations 2 and 4 predict significantly different values at 990°C; primarily because of the Reynolds number dependence of equation 4. At first sight, it appears that equation 4 is insensitive to Re_o; but the local particle Reynolds number decreases by 40 fold from 20°C to 990°C.

Very little work has been done on the measurement of Uo at elevated temperatures. Notably Singh et al(6) showed that Uo is inversely proportional to viscosity and independent of gas density. They used six different gases with silica sand up to temperatures of 890°C. Their results favour the form of equation 2. Avedesian and Davidson(7) measured Uo as a function of temperature for two different sizes of coal ash. They found that their results were well represented by equation 2 up to temperatures of 600°C. This work suggests that the more simple correlation of equation 2 is better at elevated temperatures. In the present case, either correlation may be used in view of the simplifying assumptions and the standard deviation of equation 2. The value of Uo = 4.8 cm/s may be regarded as an approximation. Of course, the best way to determine Uo is to measure it under real conditions.

The determination of the minimum fluidizing velocity leads naturally to a discussion of the two-phase theory and the bubbling bed reactor models.

The Two-Phase Theory and Reactor Models

It has long been established that most gas/solid fluidized systems exhibit phase segregation. Toomey and Johnstone(8) developed the two-phase theory which states that if the flowrate of gas to the bed is AU, then AUo calculate the minimum fluidizing velocity for the roaster bed material, is not straightforward. For instance, the fluidizing air experiences a large temperature gradient and expansion near the distributor. The air enters the tuyeres at near room temperature and reaches about 990°C in the bed. In addition, there is a change in molar volume of the gas in the bed due to chemical reaction. The local density and viscosity of the gas varies, particularly in the vicinity of the feed trajectory onto the bed. These factors, including the presence of internal cooling coils, will affect the minimum superficial velocity needed to suspend the roaster bed in a fluidized state.

As a first approximation, equations 2 and 4 were used to estimate Uo for the roaster bed using air under uniform conditions at 990°C. Equation 3 was used to calculate $\bar{d}$ from the particle size distribution of the bed overflow reported in Table IV. This assumes that the bed is perfectly mixed. Calculations give

Uo = 4.88 mm/s at 990°C from equation 2.

Uo = 3.82 mm/s at 990°C from equation 4.

The minimum fluidizing velocity was measured for a sample of bed material at 20°C. The conventional pressure drop/flowrate experiment was done using a 50 mm diam tube. The characteristic curves are shown in Figure 4 for both increasing and decreasing gas flows. As expected, the minimum fluidizing velocity is not well defined for the wide particle size distribution. It lies somewhere between 10 and 20 mm/s. It is interesting to note that both equations 2 and 4 predict the same value of Uo at 20°C; 12.6 mm/s, and this compares favourably with flows through the particulate phase and $A(U-U_0)$ flows through the bed as the bubble phase. Thus, all the gas in excess of that required for minimum fluidization passes through the bed as bubbles containing little solids and the particulate phase remains near incipient fluidization. The dense phase is in a state of violent agitation caused by the rising bubbles. There is a distribution of bubble sizes in the bed. The gas jets from the tuyeres break up into small bubbles which rapidly coalesce to form larger bubbles as they rise through the bed. The velocity of the bubbles is usually greater than the velocity of the gas in the particulate phase so that a gas cloud forms around each bubble. The bubbles exchange gas with the particulate phase by molecular diffusion, bulk flow and cloud shedding.

The early reactor models(2-10) were based on the two-phase theory. The conversion was expressed in terms of an inter-phase exchange parameter. It was assumed that the gas in the particulate phase was either in plug flow or perfectly mixed while the gas in the bubble phase was in plug flow. Orcutt et al.(14) were the first to relate the inter-phase exchange of gas between the bubble phase and the particulate phase to an effective bubble diameter. In this way, conversion could be related to the bubble diameter. Later, the model of Orcutt et al. was modified(3) to include coalescence and a distribution of bubble sizes in the bed.

The reactor models(1,11,18,19) based on bubble mechanics are a good starting point to qualitatively describe the performance of the reactor as a high temperature chemical reactor. Natesan and Philbrook(17) measured the oxidation rate of zinc sulphide particles in a batch reactor. Photomicrographs of the particles before and after oxidation showed that the particles reacted according to the shrinking core model. Their results showed that the rate determining step was chemical reaction at the surface of the outer-boundary of the unreacted sulphide core for temperatures up to 960°C. The rate of reaction was first order with respect to the oxygen concentration in the bulk of the gas stream. The work of Natesan and Philbrook can be combined with the bubble models to qualitatively describe the mechanism of roasting in the fluidized bed. The superficial gas velocity through the roaster is about 780 cm/s. According to the two-phase theory

$$\frac{U-U_o}{U} = \frac{780-5}{780} > 99\%$$

of the gas passes through the roaster in the bubble phase which contains little solids. Oxygen in the rising bubbles must transfer from the interior of the bubbles to the particulate phase by diffusion, bulk flow and cloud shedding. It must then diffuse through the particulate phase, then through the gas boundary layer surrounding a particle, and through a product oxide layer to the surface of the unreacted sulphide core. Here it reacts to produce SO_2 which counter-diffuses back to the bulk of the gas in the particulate phase. Consider a material balance on oxygen in the particulate phase. The bulk flow term based on Uo is relatively small compared with the rate of combustion. Essentially, at steady state, the rate of oxygen consumption in the particulate phase is equal to the rate of transfer from the bubble phase to the dense phase. In addition, the sulphide concentration in the particulate phase will depend on this inter-phase transfer rate. Davidson and Harrison(10) derived an expression for the inter-phase transfer rate in terms of the effective bubble diameter. This expression has been the subject of much criticism[15]; however, it does show that transfer is more rapid for small bubbles. As the bubbles grow by coalescence during their rise in the bed, the rate decreases. The conversion of sulphide to oxide can be expressed[14] as a function of the inter-phase transfer rate, the reaction velocity in the particulate phase and the ratio of superficial to minimum fluidizing velocity. The interesting feature of this bubble model[14] is that it predicts that no matter how fast the reaction in the particulate phase may be, oxygen will by-pass the bed in the bubbles and this quantity of by-passing depends on the bubble mechanics only and not on the nature of the reaction.

Although the bubble models serve a useful purpose in forming a qualitative picture of what happens in a roaster, the practical application in predicting performance or in scale-up is limited[49]. The roaster operates at over 150 times the minimum velocity for fluidization. Several workers[21,22] have shown that at these high U/Uo ratios, the bubbles rise through the bed in preferred tracks which promote de-fluidization. The fluidized bed may no longer be uniformly bubbling. This may decrease the inter-phase transfer rate and reduce the efficiency of gas/solid contact. In addition, a multi-tuyere distributor will enhance the probability of forming bubble tracks and the presence of bed coils will affect the bubble size distribution. Clearly, a priori prediction of the bubble size distribution would be difficult. The best recourse would be to start with Orcutt et al's[14] three parameter model and summarise the sulphide conversion operating data of the roaster in terms of an average equivalent bubble diameter. This average equivalent bubble diameter may bear little relation to the actual size of the bubbles in the bed, however, it may be used with caution to predict the conversion for different operating conditions in the same reactor under similar fluidization.

Elutriation Rate Constant and Particle Residence Time

Kunii and Levenspiel([illegible]) define an elutriation rate constant K^*

$$\begin{pmatrix}\text{rate of removal of solids}\\ \text{of size interval } \Delta d_i \text{ per}\\ \text{cross-sectional area of}\\ \text{bed surface, kg/m}^2\text{s}\end{pmatrix} = K^* \begin{pmatrix}\text{weight fraction of}\\ \text{bed in size inter-}\\ \text{val } \Delta d_i\end{pmatrix} \quad \ldots\ldots\ldots\ldots 5.$$

or

$$K^* = \frac{F_2\, p_2(d_i)\Delta d_i}{A\, p_b(d_i)\Delta d_i} \quad \ldots\ldots\ldots\ldots 6.$$

The residence time of particles in the size interval Δd_i in the bed is given by

$$t(d_i) = \frac{\text{weight of particles in the size interval } \Delta d_i \text{ in the bed}}{\text{flowrate of that size interval into the bed}} \quad \ldots\ldots\ldots\ldots 7.$$

or

$$t(d_i) = \frac{W p_b(d_i)}{F_1 p_1(d_i) + F_2 p_2(d_i)} \quad \ldots\ldots\ldots\ldots 8.$$

Combining equations 6 and 7

$$t(d_i) = \frac{W}{F_2 + K^* A} \quad \ldots\ldots\ldots\ldots 9.$$

Equation 9 assumes that the bed is perfectly mixed with respect to solids so that $p_i(d_i) = p_b(d_i)$. The total weight of bed material W including concentrate and calcine is estimated to be about 32 tonnes (from pressure drop measurements and stimulus-response tracer tests which will be discussed later). Equations 6 and 9 and the data in Table IV were used to calculate the elutriation rate constant and the residence time of particles in each size interval in the bed. The results of the calculation are shown in Table V; and the residence time of each size interval is plotted as a function of the arithmetic mean particle size in that interval as shown in Figure 5. It should be pointed out that the residence time is calculated for the reactor as a whole and includes the time the particles spend in the dilute suspension zone above the fluidized bed. Figure 5 shows that particles as fine as 60μm stay in the reactor for up to four hours before being elutriated or finding their way into the bed overflow. The terminal falling velocity of 60μm particles is about 150 mm/s. Compared with the superficial gas velocity in the lower hearth of 782 mm/s and in the expanded freeboard of 240 mm/s, one would normally expect these fine particles to elutriate within a short time after entering the bed.

Yagi and Aochi(4), and Wen and Hashinger(22) correlated the elutriation rate constant K^{*} as a function of the particle Reynolds number at the terminal falling velocity for a large number of data. Both correlations include the velocity factor as the difference between the superficial gas velocity and the terminal falling velocity. Clearly, in the present case, these correlations do not account for the fact that particles larger than about 100μm which have terminal falling velocities greater than the superficial gas velocity in the freeboard elutriate into the gas exit.

Furthermore, particles of about 500μm in size have terminal falling velocities of approximately 3000 mm/s; and these large particles are elutriated. Certainly, the transport disengaging height is less than 9 m which is the distance from the surface of the bed, normally 1 m deep, and the gas exit in the freeboard.

In the operation of fluidized bed zinc roasters, over 70% of the product calcine is collected as carry-over from the reactor. The suspension zone is heavily laden with particles. Bubbles errupting at the surface will eject particles into the suspension zone. The fines will accelerate and the coarse particles will decelerate as they move up. Additional momentum may be imparted to the large particles by impact with smaller fast moving particles. In this way, the coarse particles may be elutriated by momentum transfer from the smaller totally entrained particles.

A radioactive tracer test(34) was carried out to measure the residence time of the solids in the roaster. Approximately 65 tonnes of zinc concentrate containing Zn^{65} and having a total activity of about 100 millicuries was fed to the roaster over a 6-1/2 hour period at an average feed rate of 2.78 kg concentrate/s. The air flowrate was approximately 19,000 Nm^3/hr. The stimulus was a step input. At time zero, the feed was switched from the regular concentrate to the radioactive concentrate. After 6 hours and 25 minutes, the feed was switched back to the regular concentrate. Many samples were collected from the feed at the weigh belt, the bed overflow, the boiler and cyclone products where they discharged onto the main calcine conveyor belt as shown in Figure 1. The weight of the samples varied between 1-1/2 and 3-1/2 kg. The specific activity of the samples was measured using a spectrometer and a sodium iodide crystal detector. The readings in counts per minute were corrected for background and self-absorption. The results are shown in Figure 6. The average activity of the feed was 32,000 counts/min. At the end of the tracer addition, samples were taken for an additional eight hours to define the exponential decay portion of the curve. A smooth curve was drawn through the data points and extrapolated exponentially based on the decay rate data. The smooth curves were differentiated to give the hypothetical response to a pulse input or the residence time distribution of the solids in each stream. By definition, the mass average residence time is the first moment of this distribution about the origin, time zero. The following values of the mass average time were calculated from the experimental data in Figure 6.

bed overflow	5.3 hr
boiler product	1.9 hr
cyclone product	1.1 hr

The results of the tracer tests qualitatively confirm the calculations of the particle residence times in the bed as given in Table V and shown in Figure 5. For example, the cyclone product contains about 14% by weight particles in the size range -200 +325 which have a residence time in the bed of about 4-1/2 hours; and 83% in the range -325 mesh with a bed residence time of about 0.4 hours. The weighted average residence time is about one hour which compares favourably with the tracer test results. This does not include the relatively short time these fine particles spend in the waste heat boiler and the cyclone.

In summary, 10 tonnes/hr of concentrate is continuously fed to a fluidized bed weighing over 30 tonnes in which the superficial gas velocity in the lower hearth is over 150 times the minimum fluidizing velocity. The particle residence times in the reactor are of the order of hours. Whereas Nateson and Philbrook(17) have shown that the time required to completely react ZnS particles of about 0.2 mm diam bathing in 10-15% O_2 is of the order of tens of minutes at 960°C. In view of the two-phase theory and oxygen by-passing in the bubble phase, there still seems to be considerable scope for increasing the throughputs of zinc roasters while maintaining acceptable sulphide conversions and calcine quality.

CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER RESEARCH

The major incentive for further research should be to increase the throughputs of zinc roasters while maintaining a high calcine quality for leach. The reactor models based on bubble mechanics are a good starting point. They give a very good qualitative picture of the mechanism of roasting; however, the successful application of the models for predicting purposes depends on a priori estimation of the bubble size distribution in the bed. Furthermore, the bubble size distribution will depend on the distributor design and the presence of internals such as bed coils. The best recourse to develop a suitable bubble model may be through pilot plant studies. The choice of a diameter for a pilot plant roaster is important in that small beds tend to slug at high superficial gas velocities. Also, the influence of the distributor design is masked by the relatively large wall effects for small beds. The diameter of the pilot plant should be large enough such that the bubbling characteristics are similar to the full scale unit. Approximately 0.1 m^2 distributor cross-sectional area is a reasonable size for a research pilot plant.

In conjunction with developing a suitable reactor model based on bubble mechanics, the following areas of investigation are important in understanding the operation of large scale roasters.

1. Prediction of the minimum fluidising velocity of a wide particle size distribution at high temperatures.
2. The significance of the two-phase theory of fluidisation at superficial gas velocities in excess of 100 times the minimum.
3. The effect of large temperature gradients near the distributor on

bubble formation at the tuyeres.

4. The mechanism of elutriation of large particles and the retention of fine particles in a bed of wide particle size distribution. In many cases, water is sprinkled over the concentrate feed in the belief that this will agglomerate the finer particles and retain them in the bed longer.

The above pertain primarily to heterogeneous gas/solid reaction in high temperature fluidized beds. As previously pointed out, there are several important metallurgical problems which are specific to the type of concentrate roasted.

NOMENCLATURE

A	empty cross-sectional area of roaster	m^2
$\bar{d}$	the surface area mean diameter of a wide particle size distribution. Defined by equation 3	m
d_i	the arithmetic average particle diameter in size interval i	m
Δd_i	size interval i	m
F_1	flowrate of calcine leaving the roaster from the bed overflow	kg/s
F_2	flowrate of calcine leaving the roaster from the gas exit	kg/s
g	acceleration of gravity	m/s^2
K^*	elutriation rate constant defined by equations 5 and 6	kg/m^2s
$p(d_i)$	particle size distribution; subscripts 1 = in the bed overflow, 2 = in the off-gas leaving the roaster, b = in the fluidised bed	m^{-1}
Re_p	local particle Reynolds number defined by $Re_p = \rho_g U d_i/\mu$	--
$t(d_i)$	residence time of particles of size d_i in the reactor	s
U	superficial gas velocity based on A and at the mean bed temperature	m/s
U_o	= U at incipient fluidisation	m/s
W	weight of fluidized bed	kg
μ	viscosity of gas	kg/ms
ρ	density . subscripts g = gas s = solid	kg/m^3

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TABLE II

MATERIAL FLOWRATES FOR AN OPERATING DAY JULY 1974

Solids

Stream	kg./s
feedrate of concentrate	2.66
bed overflow	0.52
boiler product (water cooled (settling chamber))	1.24
cyclone product	0.26
precipitator product	0.05
elutriation rate	Σ 1.55
total calcine production rate	Σ 2.07

Gas

Stream	Composition % by volume O_2	N_2	H_2O	SO_2	Flowrate Nm^3/hr
inlet air	21.	79.	–	0	19364
off-gas	4.0	77.3	7.8	10.9	19771
	100 %				
off-gas excluding H_2O :	4.3	83.9	–	11.8	$19771 \times \frac{92.2}{100} = 18229$

Figures not consistent with the feed of solids, i.e. too much O_2 used up.

Water addition to concentrate ~ $180 \times 10^{-6} m^3/s$ = ·180 Kg./s

= 7.2 %

TABLE 7

TYPICAL CHEMICAL ANALYSIS OF ZINC CONCENTRATE

% by weight dry basis

53.4 Zn		∼ 1.5 SiO_2
33.0 S	97.1	∼ 0.4 MgO
10.7 Fe		∼ 0.3 CaO
0.6 Cu		∼ 0.1 Al_2O_3
0.3 Cd		∼ 0.1 Mn

Including trace quantities of minor elements.

Typical moisture content 5.8%

TABLE III

INTENSIVE PROPERTIES OF GAS

Stream	Static Gauge Pressure mm Hg	Temperature °C	Dust Loading mg /Nm³
wind box	116	25	0
freeboard	0.2	990	282 x 10³
boiler exit	0	320	56.4 x 10³
cyclones exit	-4.8	315	9.1 x 10³
precipitator exit	-7.1	310	120

Pressure drop across the distributor = 40 mm Hg estimated by measurement on a single tuyere.

TABLE IV

PARTICLE SIZE DISTRIBUTION ANALYSIS FOR MONTHLY COMPOSITES JULY 1974

Tyler Mesh	Opening Range mm.	% by weight in each size fraction						
		Concentrate Feed	Bed Overflow	Total Elutriated Solids[x]	Boiler Product[x]	Cyclones Product	Precipitator Product	Total Calcine
-28+35	-0.599+0.420	0.3	9.2	0.9	1.1	0.1	0.0	3.3
-35+65	-0.420+0.210	3.6	10.7	4.2	5.2	0.1	0.0	5.5
-65+100	-0.210+0.149	9.1	17.0	2.3	2.8	0.2	0.0	6.0
-100+150	-0.149+0.105	9.8	18.9	3.3	4.0	0.4	0.1	7.2
-150+200	-0.105+0.074	12.8	26.8	7.3	8.6	2.6	0.2	12.7
-200+325	-0.074+0.044	13.3	12.2	11.7	11.7	13.8	0.8	11.8
-325	-0.044	51.1	5.2	70.3	66.6	82.8	98.9	54.0

x calculated by material balance

TABLE V

ELUTRIATION RATE CONSTANT AND PARTICLE RESIDENCE TIME IN THE BED*

Tyler Mesh	d_i mm	K^* kg/m^{2}s × 10^3	$\tau(d_i)$ s × 10^{-3}
-28 +35	0.510	4.71	48.3
-35 +65	0.315	18.9	28.7
-65 +100	0.180	4.37	49.1
-100 +150	0.127	8.26	41.2
-150 +200	0.090	13.1	34.4
-200 +325	0.059	46.5	16.0
-325	0.022	681	1.48

* Calculated from data in Table IV

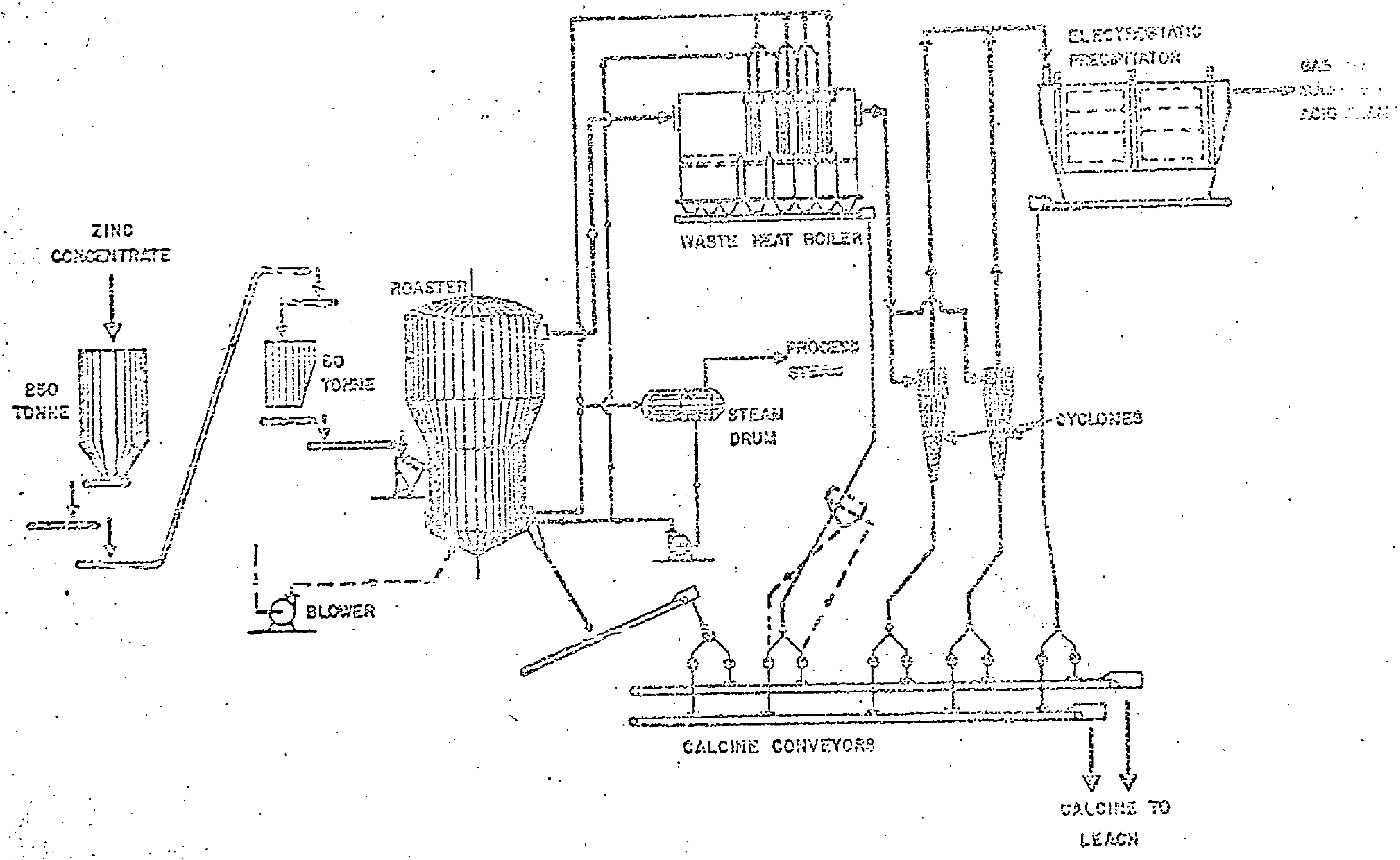


FIGURE – I – The Roaster Plant Flowsheet.

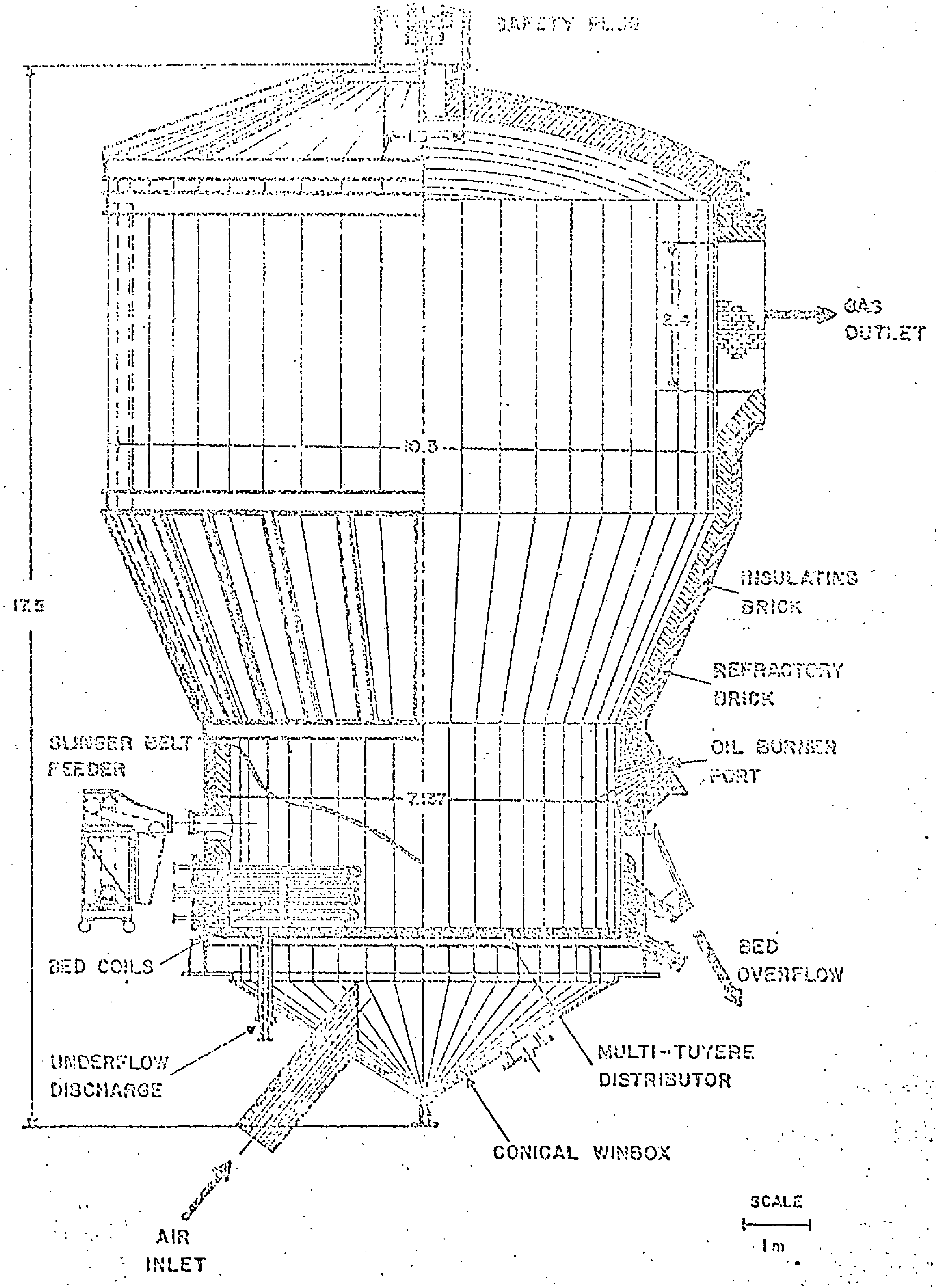


FIGURE – 2 – Lurgi Turbulent Layer Roaster

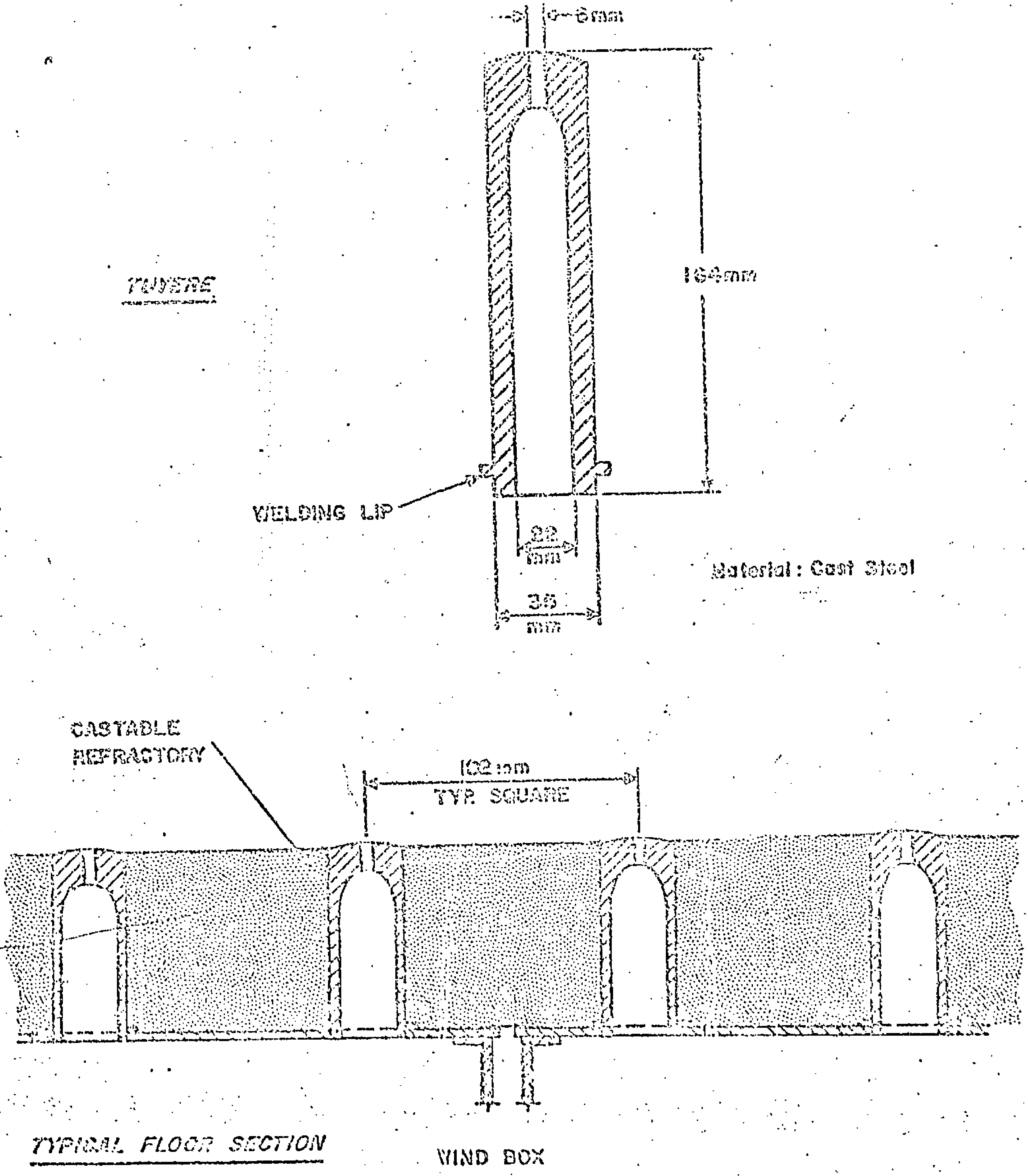


FIGURE – 3 – The Multi-Tuyere Distributor

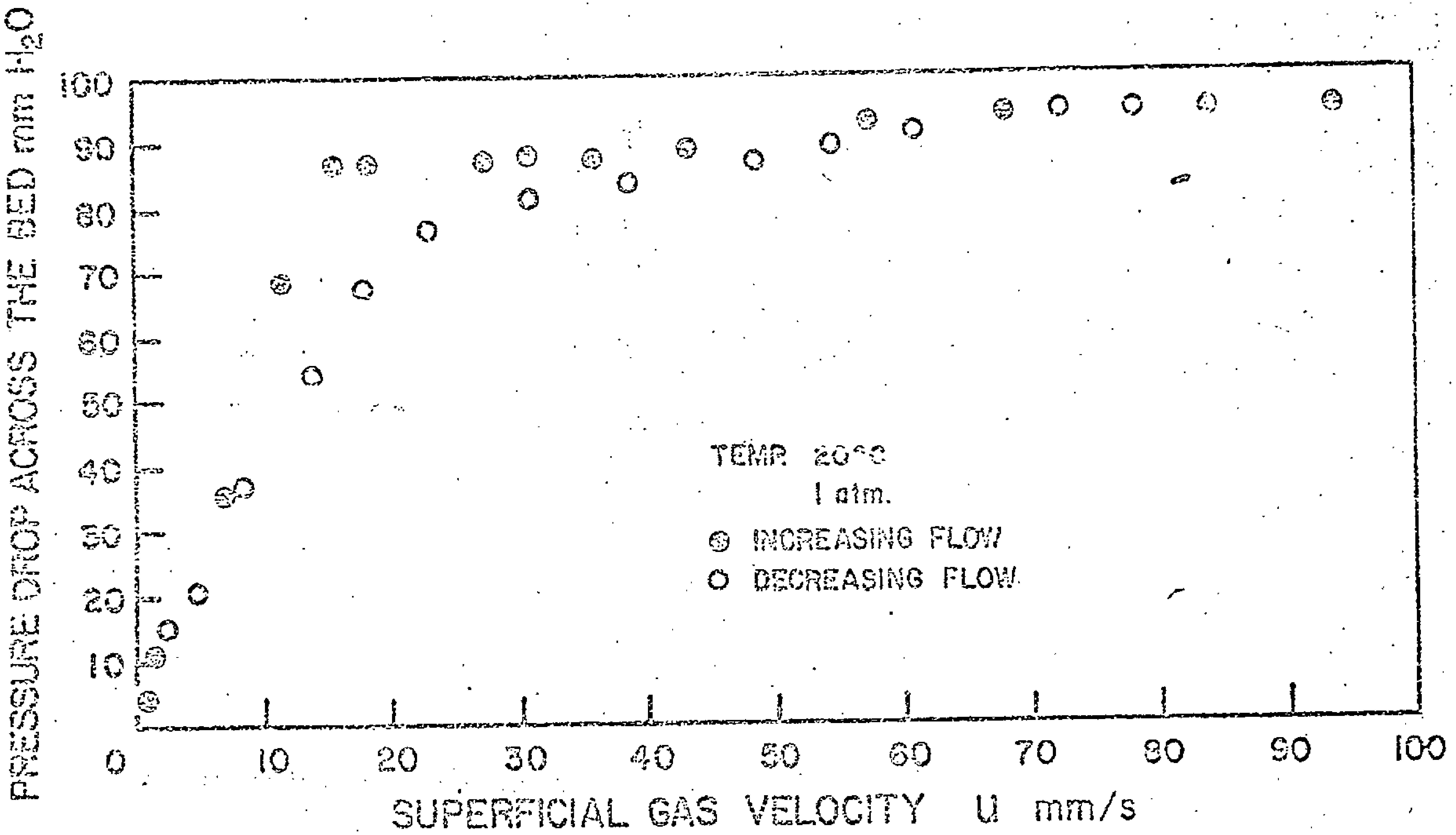


FIGURE – 4 – Determination of the Minimum Fluidizing Velocity for the Roaster Bed Material.

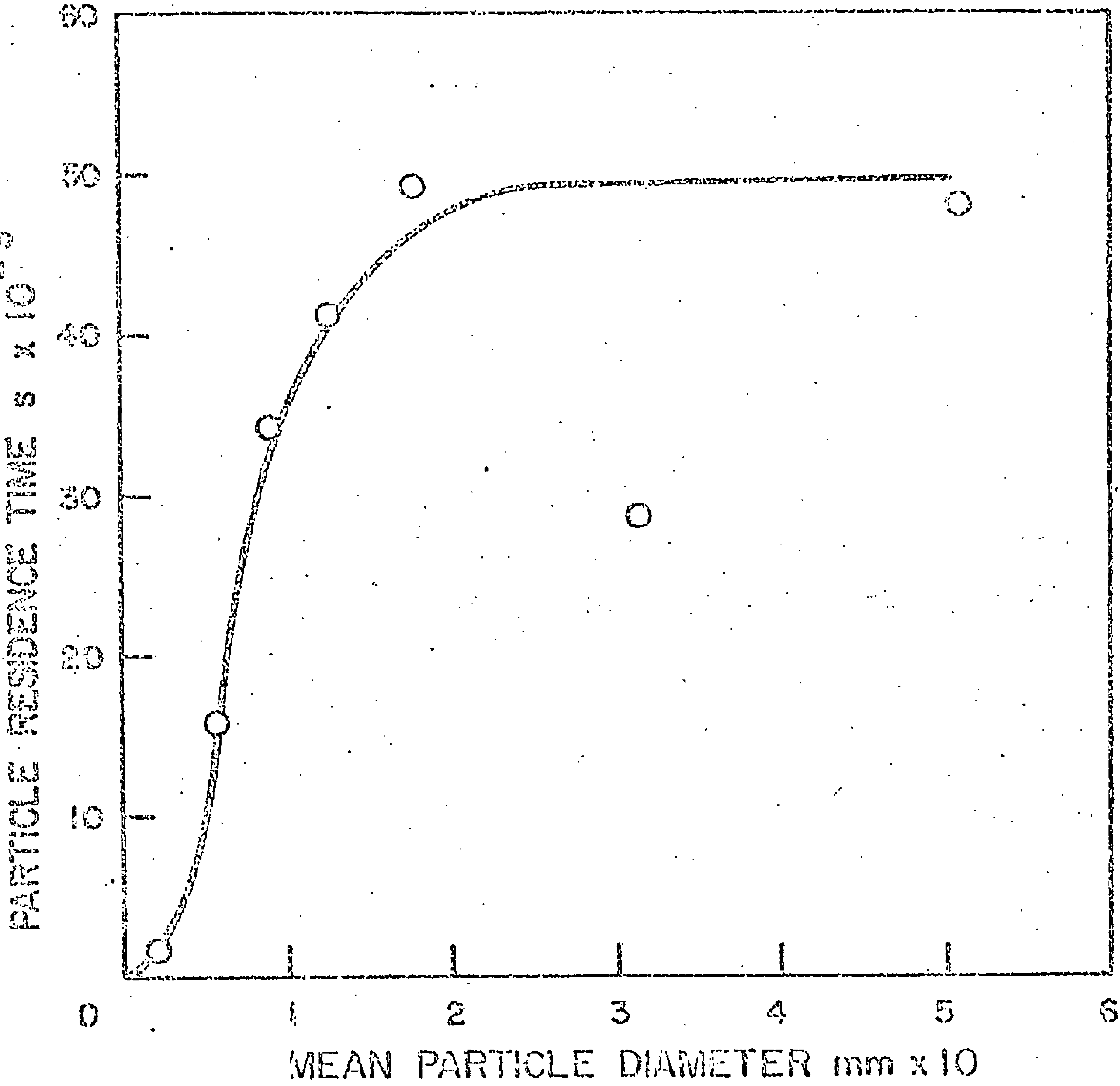
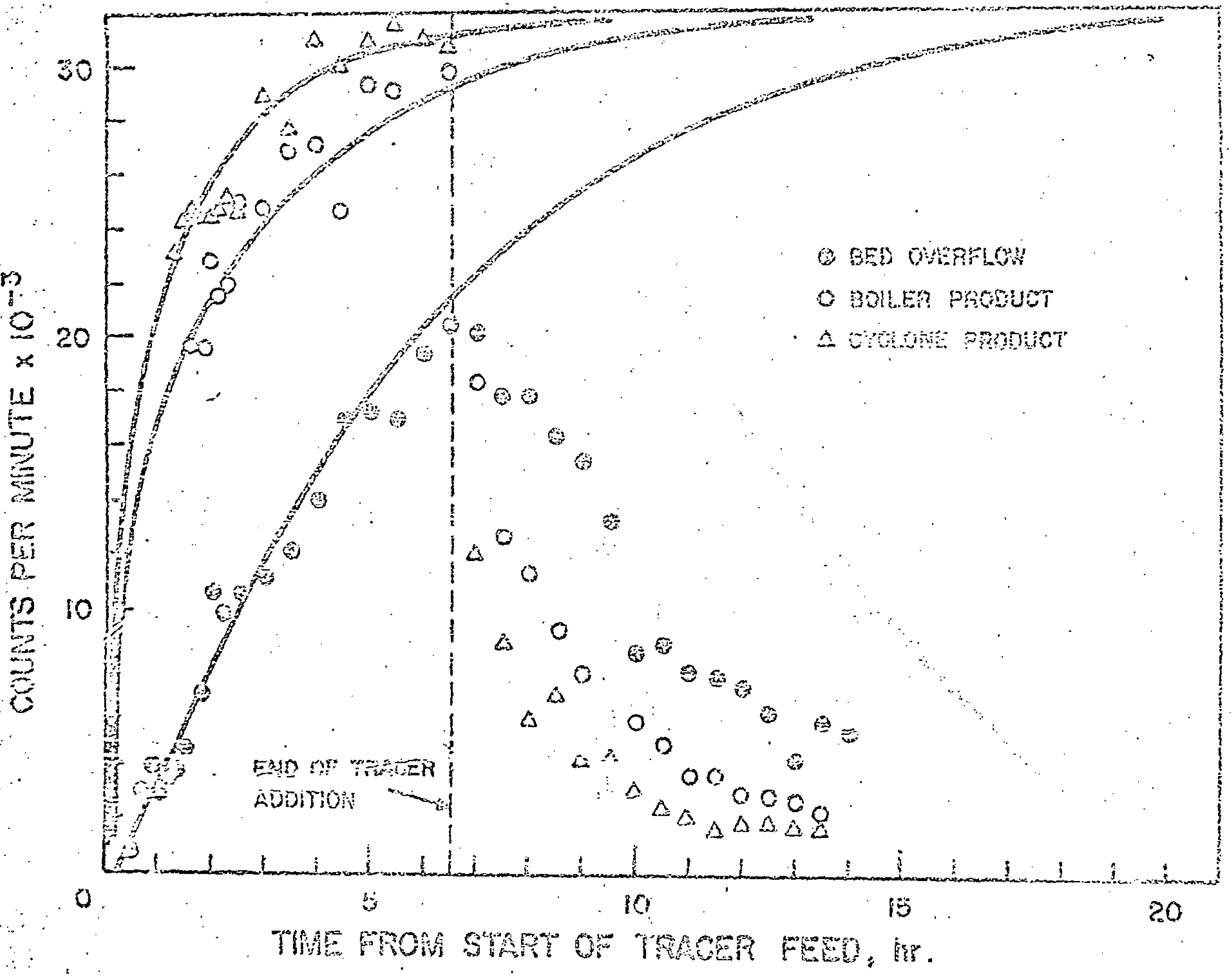


FIGURE-5-Residence Time of Particles In The Reactor Bed.



[illegible] Curves for Solids Streams for A Step Input of Tracer to the Reactor.

Oxidation of Zinc Sulfide in a Fluidized Bed

Y. FUKUNAKA, T. MONTA, Z. ASAKI, AND Y. KONDO

Kinetics of oxidation of ZnS particles in a batch-type fluidized bed were studied at temperatures between 800 and 910°C. A two-phase model was employed for the fluidized bed, and the partial pressure of oxygen and the gas-film mass transfer coefficient on the particle surface were separately evaluated in gas bubbles and in the emulsion phase. The calculated fractional reaction coincided well with the experimental results. The difference in O_2 partial pressure between gas bubbles and emulsion phase was found to be fairly large especially under the vigorous fluidizing condition. Furthermore, it was shown from the mathematical model that the reaction of ZnS particles in the gas bubbles is negligible because of the extremely low solid concentration and that the overall rate of reaction in the emulsion phase is virtually controlled by the rate of gas-film mass transfer at higher temperature. The resistance of interfacial reaction within the particle also becomes significant when the temperature is lowered.

MANY experimental studies[1-12] have been conducted on the oxidation kinetics of ZnS. Zinc sulfide of stoichiometric composition is stable in the unreacted inner core of the particle during the oxidation, and the kinetic analysis is facilitated as compared with other nonstoichiometric sulfide minerals. It can be concluded from the previous work[2-9] that the interfacial reaction is the rate-determining step below about 700°C and inward diffusion of O_2 gas through the outer oxide shell determines the overall rate above this temperature.

A few experimental studies[10-12] were conducted on the oxidation of ZnS particles in fluidized bed reactors, in which the uniform-phase model for fluidized bed was employed. Along with the development of the fluidization technique, however, it was recognized that an aggregative gas-solid fluidized bed is composed of gas bubbles and an emulsion phase and that the behavior of fluidized beds is closely related to the rising gas bubbles.[13] It is also recognized that the excess fluidizing gas above that required for incipient fluidization rises through the bed in the form of gas bubbles in which the fraction of solid particles is extremely low.[14-24] The solid particles in the gas bubbles are supposed to move at the terminal velocity. The emulsion phase is regarded to be very similar to the state of incipient fluidization and the flow rate of gas in the emulsion phase is presumed to be at the superficial velocity in incipient fluidization. In the kinetics of fluidized bed roasting of sulfide particles in which the mass transfer through the gas-film on the particle surface may play an important role, it is preferable to employ the two-phase model and to evaluate separately the gas-film mass transfer coefficient of particles suspended in gas bubbles and in the emulsion phase.

The oxidation kinetics of ZnS particles in a batch-type fluidized bed are studied in this work. In order to simplify the analysis, the reaction was carried out under essentially isothermal conditions. Zinc sulfide particles were mixed with a large amount of fused alumina particles, and the mixture was fed to the fluidized bed in order to minimize the temperature rise of the bed during the course of reaction.

MATHEMATICAL MODEL

In a batch-type fluidized bed, the fractional reaction of each fluidized particle is presumed to be uniform when the particles of the same size are fed to the reactor at the same time. The following assumptions were made on the oxidation of a single ZnS particle in the fluidized bed:

1) The unreacted core model is employed, and the sequential steps of i) mass transfer of O_2 gas through the gas-film on the particle surface, ii) inward diffusion of O_2 gas through the outer oxide shell, and iii) interfacial reaction are presumed.

2) The interfacial reaction between O_2 gas and the inner ZnS core is assumed to be a first-order irreversible reaction with respect to the O_2 partial pressure.

3) The temperature profile within the particle is uniform because of the small particle size, *i.e.* resistance to heat transfer within the particle is neglected.

4) The temperature difference across the gas-film on the particle surface is also assumed negligible.

The oxidation of ZnS is expressed as

$$ZnS + \frac{3}{2}O_2 = ZnO + SO_2 \qquad [1]$$

$\Delta H = -106$ kcal/mol ZnS (600 to 1000°C).

The unreacted core model is schematically illustrated in Fig. 1. Based on the above-mentioned assumptions, the rate equation for each sequential step is

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$$\frac{dn_{O_2}}{dt} = 4\pi r_o^2 k_g (C - C_s)$$

$$= 4\pi \frac{r_o r_i}{r_o - r_i} D_e (C_s - C_i)$$

$$= 4\pi r_i^2 k_r C_i. \qquad [2]$$

Then the overall rate equation is

$$\frac{dn_{O_2}}{dt} = 4\pi r_o^2 k C \qquad [3]$$

where

$$\frac{1}{k} = \frac{1}{k_g} + \frac{r_o^2}{D_e r_i}\left(1 - \frac{r_i}{r_o}\right) + \frac{1}{k_r}\left(\frac{r_o}{r_i}\right)^2. \qquad [4]$$

The rate of outward diffusion of SO_2 gas through the oxide shell is related to the inward diffusion rate of O_2 gas by the stoichiometric relationship of Eq. [1]. However, from the assumption of irreversible interfacial reaction, knowledge of the diffusion rate of SO_2 is not necessary for deriving the overall rate equation.

The fluidizing air stream blown into the bed from the bottom is divided into two portions: a portion of fluidizing gas flows through the emulsion phase at a velocity of u_{mf} and the remainder rises through the bed in the form of gas bubbles whose volume fraction in the bed is δ.[21] A mass balance with regard to the fluidizing gas yields the following equation:[26]

$$u_o = \delta u_B + (1 - \delta) u_{mf}. \qquad [5]$$

The above assumption implies that the void fraction in the emulsion phase is maintained at ϵ_{mf}. Thus the bed expansion above the bed height at incipient fluidization is presumed to be caused by the presence of gas bubbles, and we have

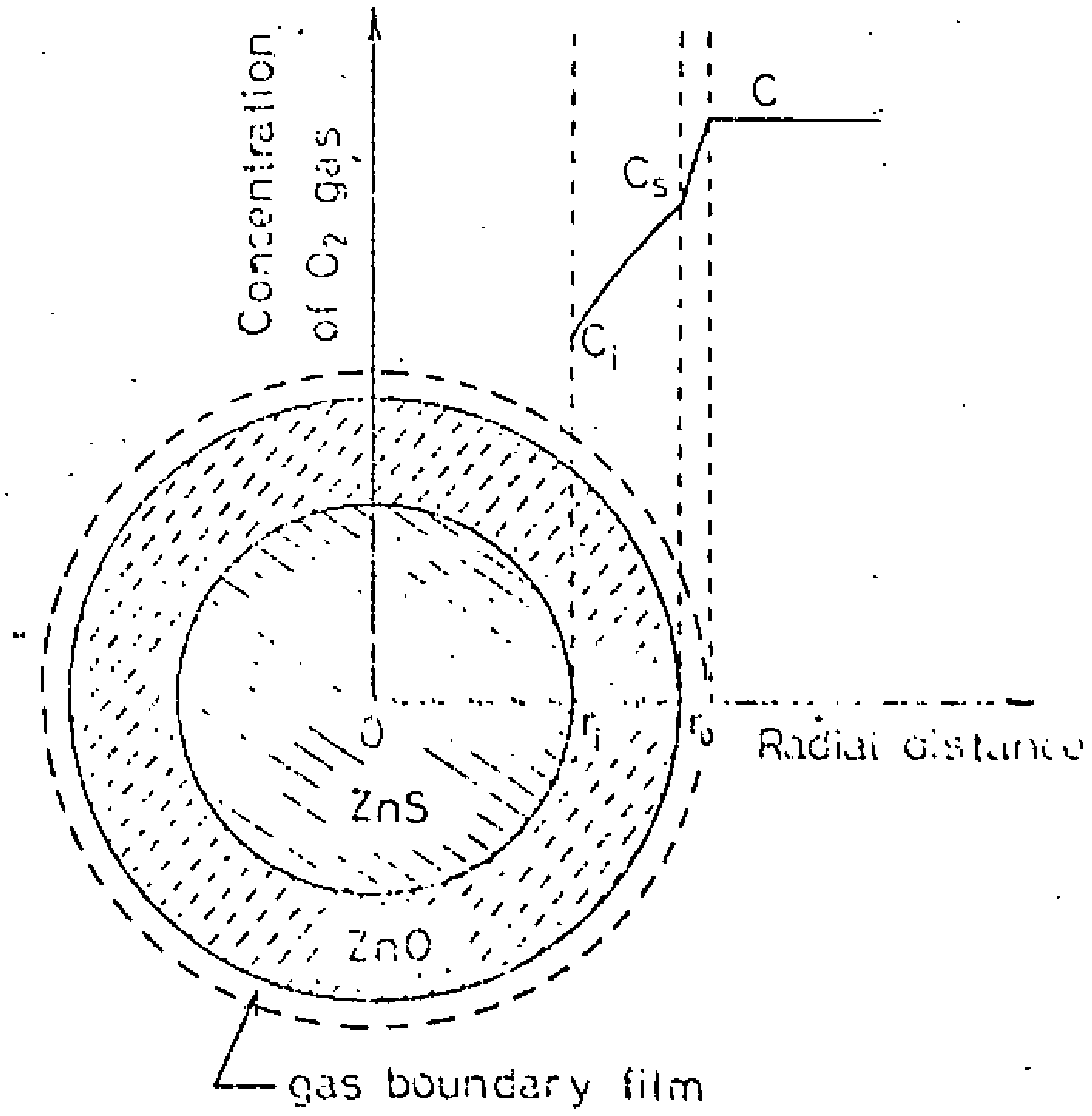

Fig. 1—Shrinking core model.

$$\delta = 1 - (L_{mf}/L_f). \qquad [6]$$

Furthermore, the absolute velocity of gas flow in the emulsion phase is

$$u^E = u_{mf}/\epsilon_{mf}. \qquad [7]$$

In order to derive the mass balance equation for O_2 gas in the fluidized bed, the following two assumptions were made.

1) The fluidized particles are completely mixed within the bed, and the upward gas flow through each phase is in plug flow.

2) The flow rate of fluidizing gas is constant along the height of bed; the reduction of gas volume due to the oxidation of ZnS particles is only a few percent even when the reaction is completed instantaneously.

Based on these assumptions, we have the following O_2 gas mass balance equation for the gas bubbles:

$$S\delta\epsilon^B \frac{\partial C^B}{\partial t} = -S\delta u_B \frac{\partial C^B}{\partial z} - \frac{N^B}{L_f} 4\pi r_o^2 k^B C^B - S\delta K_{BE} \times (C^B - C^E) \qquad [8]$$

and for the emulsion phase

$$S(1-\delta)\epsilon_{mf} \frac{\partial C^E}{\partial t} = -S(1-\delta) u_{mf} \frac{\partial C^E}{\partial z} - \frac{N^E}{L_f} 4\pi r_o^2 k^E C^E + S\delta K_{BE}(C^B - C^E). \qquad [9]$$

The numbers of ZnS particles in gas bubbles and in the emulsion phase, N^B and N^E, appearing in Eqs. [8] and [9] are given by

$$N^B = \frac{3 S L_f \delta \gamma \phi}{4\pi r_o^3} \qquad [10]$$

and

$$N^E = \frac{3 W_{ZnS}}{4\pi r_o^3 \rho_{ZnS}} - N^B \qquad [11]$$

respectively. The accumulation terms on the left-hand side of Eqs. [8] and [9] can be neglected in the calculation within an infinitesimal time interval. Then, the following linear simultaneous ordinary differential equations are obtained:

$$\frac{dC^B}{dz} = -(a + b)C^B + bC^E \qquad [12]$$

$$\frac{dC^E}{dz} = -(c + d)C^E + dC^B \qquad [13]$$

where

$$a = \frac{4\pi r_o^2 k^B N^B}{S\delta u_B L_f}, \qquad b = \frac{K_{BE}}{u_B}$$

$$c = \frac{4\pi r_o^2 k^E N^E}{S(1-\delta) u_{mf} L_f}, \qquad d = \frac{\delta K_{BE}}{(1-\delta) u_{mf}}.$$

By applying the Laplace transformation technique to Eqs. [12] and [13], we have

$$C^B = \frac{C_o}{\alpha - \beta}\{(\alpha + b + c + d)\exp(\alpha z) - (\beta + b + c + d)\exp(\beta z)\} \qquad [14]$$

$$C^E = \frac{C_o}{\alpha - \beta}\{(a + b + \alpha + d)\exp(\alpha z) - (a + b + \beta + d)\exp(\beta z)\} \qquad [15]$$

where C_o is the concentration of O_2 gas at the bottom of the fluidized bed, and the parameters α and β are the solutions of the following equation:

$$t^2 + (a + b + c + d)t + (ac + ad + bc) = 0. \qquad [16]$$

From Eqs. [14] and [15], the concentration profiles of O_2 gas in the gas bubbles and in the emulsion phase at any given time are calculated. By substituting the obtained values of C^B and C^E in Eq. [3], the consumption of O_2 gas in a time interval Δt is calculated. In gas bubbles, it is

$$(n_{O_2})^B = \int_0^{L_f} \left(\frac{dn_{O_2}}{dt}\right)_B (\Delta t)\, \frac{N^B}{L_f}\, dz \qquad [17]$$

and in the emulsion phase, we have

$$(n_{O_2})^E = \int_0^{L_f} \left(\frac{dn_{O_2}}{dt}\right)_E (\Delta t)\, \frac{N^E}{L_f}\, dz. \qquad [18]$$

The consumption of O_2 gas in the whole fluidized bed is

$$n_{O_2} = (n_{O_2})^B + (n_{O_2})^E. \qquad [19]$$

By using n_{O_2}, the time variation of r_i is calculated by the equation

$$\frac{4\pi r_0^2 (\text{pct S})}{100 M_S} \cdot \frac{3W_{ZnS}}{4\pi r_0^3} \cdot \frac{dr_i}{dt} = \frac{2}{3} n_{O_2}. \qquad [20]$$

Furthermore, the fractional reaction of particles, x, was calculated by using the geometric relationship

$$x = 1 - \left(\frac{r_i}{r_o}\right)^3. \qquad [21]$$

The calculation was iterated with a time interval of 1.5 s starting from $x = 0$ at $t = 0$.

EXPERIMENTAL

The experimental arrangement is schematically illustrated in Fig. 2. Nitrogen gas and air were supplied from a nitrogen gas cylinder and a small compressor, respectively. Dried air and nitrogen gas passed through a change-over valve and were sent to the fluidized bed reactor or exhausted into the atmosphere through a mercury reservoir, respectively. By adjusting the depth of glass tube immersed in the reservoir, both gases were maintained at the same pressure.

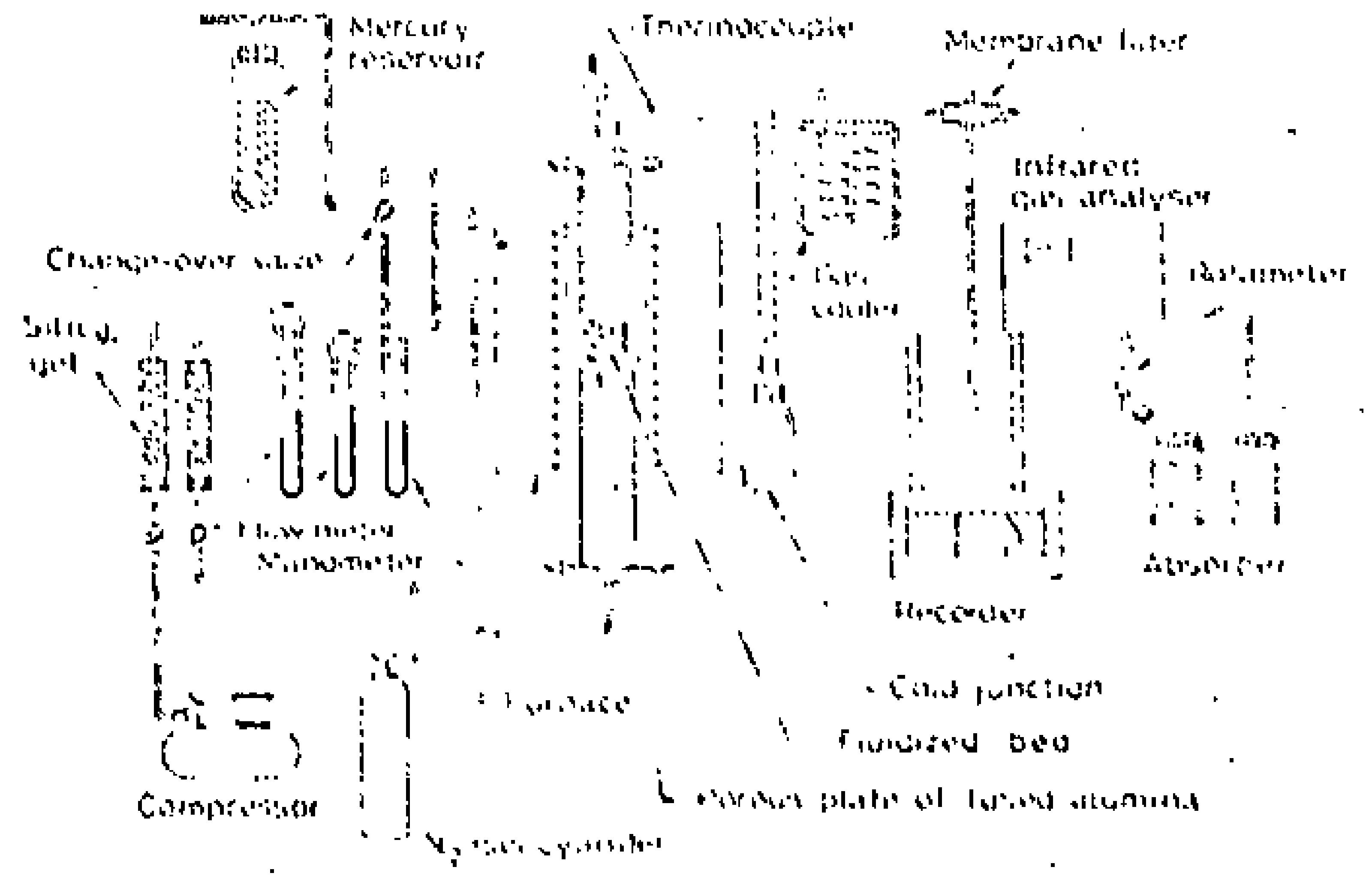

Fig. 2—Schematic representation of the experimental apparatus.

The fluidized bed reactor was constructed by welding a stainless steel tube, 76.3 mm OD, 62.3 mm ID and 400 mm long, to the end of another stainless steel tube, 60.5 mm OD, 52.7 mm ID and 600 mm long. A distributor was supported on the upper edge of an inner stainless steel tube of 60.5 mm OD which was inserted from the bottom of the reactor. The distributor was a porous sintered alumina plate, 60.5 mm in diam and 5 mm thick. The temperature of the fluidized bed was measured with a Pt-Pt Rh thermocouple which was installed in the bed 3 cm high above the distributor.

The exit gas from the reactor was sent to a train of a gas cooler maintained at 0°C and a membrane filter in order to remove moisture and entrained dust. After passing through a rotameter, the SO_2 concentration was continuously measured and recorded by a Shimadzu-Hartmann Braun infrared gas analyzer.

Zinc sulfide particles were prepared by screening a flotation concentrate of sphalerite. The chemical assay of the ZnS particles is shown in Table I. The weight fraction of ZnS in the flotation concentrate was calculated to be 0.83. As seen in Eq. [1], the oxidation of ZnS is highly exothermic. In order to obtain essentially isothermal condition, 1.5 g or less of ZnS particles of 60 to 100 mesh size were mixed with 200 g of fused alumina particles of the same size and the mixed particles were fluidized. The density of the fused alumina particles was 4.3 g/cm³ which is very close to the 4.1 g/cm³ density of the ZnS particles. The temperature rise of the fluidized bed was less than 5°C throughout the study. Experiments were carried out at temperatures between 800 and 910°C, and the flow rate of fluidizing air was chosen at about 1.5, 3, 5 and 10 times u_{mf}.

The rate of oxidation of ZnS particles was determined from the SO_2 concentration in the exit gas. After leaving the fluidized bed, the gas passed through the upper empty portion of the reactor, the gas cooler, the membrane filter and the rotameter before it entered the absorption cell of the gas analyzer. It was therefore necessary to correct the measured SO_2 concentration for the path volume between the fluidized bed and gas analyzer. Assuming that the path volume is a linear combination of volumes of plug flow and backmix flow, the former contributes to a delay in response at the gas analyzer. For the volume of backmix flow, V, we have

$$v(C_f - C_a) = \frac{d}{dt}(VC_a). \qquad [22]$$

From this equation,

$$C_f = C_a + \theta \frac{dC_a}{dt} \qquad [23]$$

where the mean residence time, θ, is defined as V/v. Integration of Eq. [23] yields

Table I. Chemical Assay of ZnS Concentrate

Zn, Pct	Fe, Pct	S, Pct	SiO_2, Pct
55.7	5.5	30.7	3.7

$$\ln\left(1 - \frac{C_a}{C_f}\right) = -\frac{t}{\theta}. \qquad [24]$$

Prior to the roasting experiments, the mean residence time was measured. An amount of 200 g of fused alumina particles was fluidized at 800°C in a nitrogen gas stream of various flow rates. Then SO_2 gas at a constant flow rate was added as a step-input signal, and the time variation of the SO_2 concentration was measured. The mean residence time θ was calculated from the slope of the linear portion of a plot of $\ln(1 - C_a/C_f)$ *vs* t, and it was found to be represented by the following empirical equation:

$$\theta = \frac{483.1}{v}. \qquad [25]$$

By using Eqs. [23] and [25], C_f was obtained from the measured SO_2 concentration. The fractional reaction, x, was obtained from the curve of corrected SO_2 concentration *vs.* reaction time by graphical integration. The fractional reaction thus obtained is summarized in Table II and plotted in Fig. 3. It is seen in this figure that the rate of reaction increases with the bed temperature and with the flow rate of air.

DISCUSSION

In order to calculate the fractional reaction from Eqs. [3] through [21] and to compare them with the experimental ones, it is necessary to estimate the parameters which appear in these equations. They are classified into two groups: the parameters, δ, u_B, K_{BE} and γ concern the behavior of the fluidized bed, and the other parameters k_g, D_e and k_r concern the overall rate of oxidation of ZnS particles. The volume fraction of gas bubbles in the fluidized bed, δ, was estimated from Eq. [6]. The rising velocity of gas bubbles, u_B, is related to the bubble diameter, d_B, by:[25]

$$u_B = u_o - u_{mf} + 0.711\,(g d_B)^{1/2}. \qquad [26]$$

The value of d_B in this equation was estimated from the following empirical equation proposed by Kobayashi:[16]

$$d_B = 1.4\, d_p \rho_p \left(\frac{u_o}{u_{mf}}\right) z. \qquad [27]$$

Although d_B varies with the height above the bottom of the fluidized bed, the mean bubble diameter, $\bar{d}_B$, at $z = L_f/2$ was calculated from Eq. [27], and u_B was

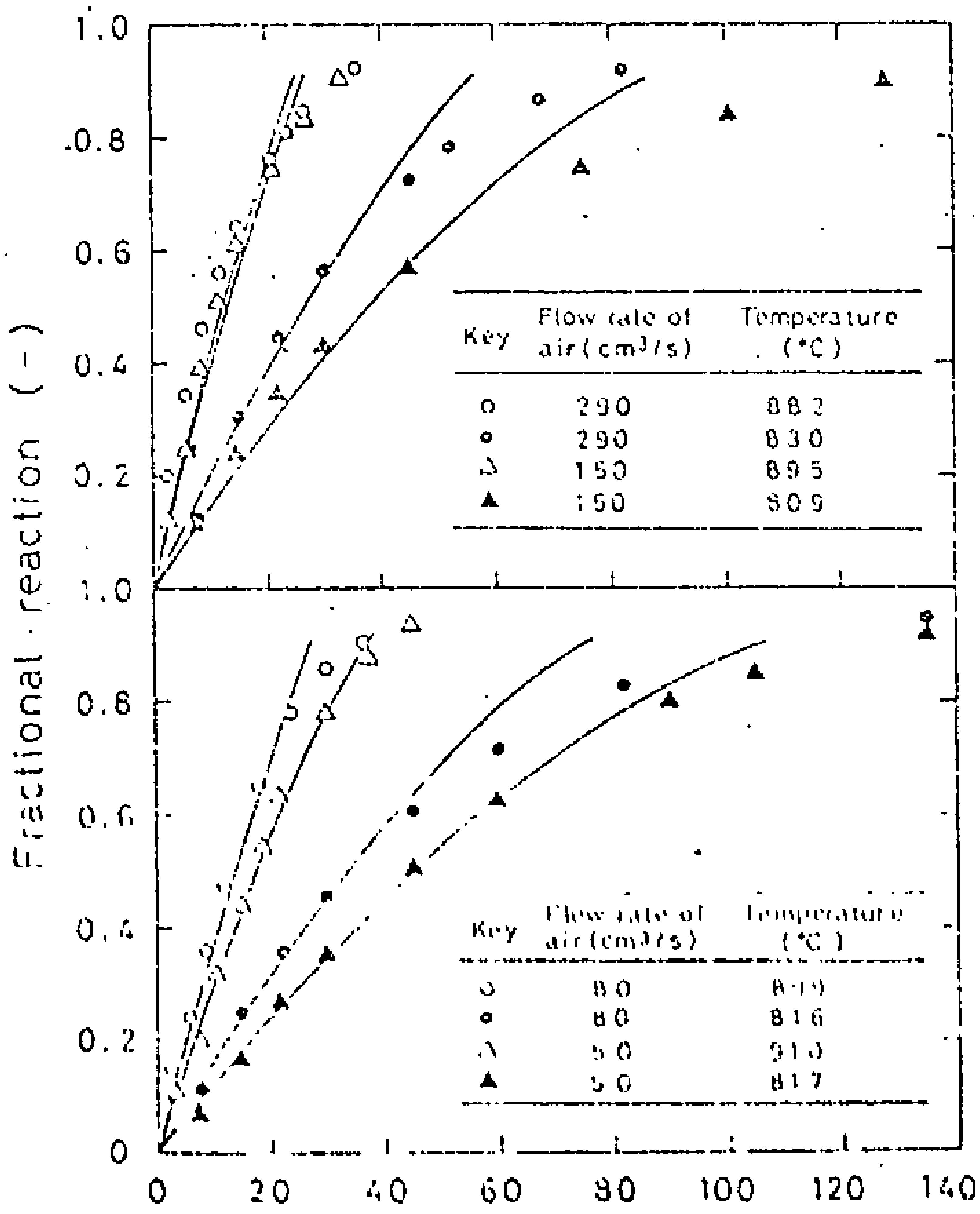

Fig. 3—Fractional reaction *vs* reaction time.

Table II. Fractional Reaction

Flow Rate of Air	Bed Temperature			
	882°C		830°C	
	t (s)	x (–)	t (s)	x (–)
290 (cm³/s)	3	0.200	8	0.127
	6	0.346	15	0.308
	9	0.464	22	0.449
	12	0.560	30	0.564
	15	0.642	45	0.728
	21	0.764	52	0.786
	24	0.810	68	0.869
	27	0.848	82	0.919
	36	0.924		
	895°C		809°C	
	t (s)	x (–)	t (s)	x (–)
150 (cm³/s)	3	0.122	8	0.112
	6	0.251	15	0.238
	9	0.393	22	0.345
	12	0.512	30	0.432
	15	0.610	45	0.570
	21	0.749	75	0.745
	27	0.845	101	0.840
	33	0.906	128	0.901
	899°C		816°C	
	t (s)	x (–)	t (s)	x (–)
80 (cm³/s)	3	0.151	8	0.115
	6	0.241	15	0.249
	9	0.359	22	0.362
	12	0.473	30	0.459
	18	0.652	45	0.612
	24	0.752	60	0.720
	30	0.859	82	0.832
	36	0.906	135	0.948
	910°C		817°C	
	t (s)	x (–)	t (s)	x (–)
50 (cm³/s)	4	0.111	8	0.072
	8	0.200	15	0.168
	11	0.320	22	0.268
	15	0.442	30	0.354
	19	0.545	45	0.508
	22	0.638	60	0.627
	30	0.785	90	0.799
	38	0.878	105	0.854
	45	0.938	135	0.923

approximated to be constant within the bed. It was obtained by substituting d_B in Eq. [26]. The gas exchange coefficient, K_{BE}, was estimated from the following empirical relationship proposed by Kobayashi:[15]

$$K_{BE} = \frac{11}{d_B} . \quad [28]$$

Furthermore, Hiraki *et al*[27] reported that the concentration of particles in gas bubbles, ρ_B, is between 6 and 9 kg/m³. From the value of ρ_B and the bulk density of the particles, γ was calculated to be between 0.004 and 0.006. A γ value of 0.005 was therefore employed. It was found that a variation in the γ value of ±20 pct scarcely affects the calculated fractional reaction and concentration profile of O_2 gas in the bed.

Among the three kinetic parameters, the value of k_g is different in the gas bubbles and in the emulsion phase. The gas flow within the gas bubbles is a vigorous circulating flow, and it was presumed that the relative velocity of solid particles is maintained at the terminal velocity.[17] The gas-film mass transfer coefficient in gas bubbles, k_g^B, was estimated by using the Ranz-Marshall equation:[16]

$$\frac{k_g^B d_p}{D} = 2.0 + 0.6\left(\frac{\nu}{D}\right)^{1/3}\left(\frac{\rho_g u_t d_p}{\mu}\right)^{1/2} . \quad [29]$$

From this equation, the numerical value of k_g^B was calculated to be about 8×10^2 cm/s under the experimental conditions. On the other hand, the numerical value of k_g^E in the emulsion phase was estimated from previous work,[19,20] and was found to be between 2.1 and 6.5 cm/s; a value of 4.2 cm/s was therefore employed in the fractional reaction calculations.

The effective diffusivity of O_2 gas in the outer oxide shell of particles was estimated from the Wakao-Smith equation[21]

$$D_c = \epsilon^2 D \quad [30]$$

where

$$\frac{1}{D} = \frac{1}{D_M} + \frac{1}{D_K} .$$

The molecular diffusivity, D_M, and the Knudsen diffusivity, D_K, were calculated by the Chapman-Enskog equation[22] and by the equation[23]

$$D_k = 9700 r_e \sqrt{\frac{T}{M_{O_2}}} \quad [31]$$

respectively. The porosity, ϵ, and mean pore radius, r_e, of the oxide which was prepared by the oxidation of ZnS particles were measured by means of the mercury penetration technique. The calculated effective diffusivity was 0.048 and 0.378 cm²/s for the oxide particles obtained at 800 and 900°C, respectively. The significant temperature variation of this diffusivity ... presumed to be due to the difference in the pore structure of the oxide particles.

The overall rate of oxidation of ZnS particles was measured by many workers in the past.[1-9] In order to estimate the rate constant of interfacial reaction from the reported experimental data, the reported overall rate at the start of reaction was corrected for the resistance of gas-film mass transfer on the particle surface. In this correction, first-order reaction was presumed with respect to the O_2 partial pressure. The k_r values obtained are shown in Fig. 4. It is seen in this figure that the estimated activation energies are similar among the workers, though a wide variation of k_r values is observed. This variation is presumed to be caused by the difference in shape, surface roughness, reactivity, *etc* of the ZnS particles used. Since it was difficult to choose the proper value of k_r from this figure, the theoretically calculated values of fractional reaction were superimposed on the experimentally measured ones, and it was attempted to obtain a close fit between them by varying the k_r value.

The values of parameters employed in the fractional reaction calculations are summarized in Table III. The calculated fractional reaction is shown as the solid lines in Fig. 3. As seen in this figure, each theoretical curve coincides well with the respective experimental result below a fractional reaction of about 0.7. It is also seen in this figure that the fit becomes rather poor when the flow rate of air is increased at about 900°C. Under this experimental condition, the overall rate is very high and the reaction is completed within a minute. Then, the experimental errors involved in the measurement and correction of SO_2 concentration in the exit gas may give rise to the poor fit. Moreover, it is known[24] that the breakup of rising gas bubbles and the stalactites of falling particles in the gas bubbles become significant under the conditions of vigorous fluidization which is realized at higher flow rates of air. In addition, the circulating gas through the cloud on the bubble surface markedly affects the overall rate of reaction.[24] These phenomena of rising gas bubbles which are not taken into consideration in the above two-phase model may also be another cause of the poor fit.

Values of k_r are plotted against $1/T$ in Fig. 4. It is

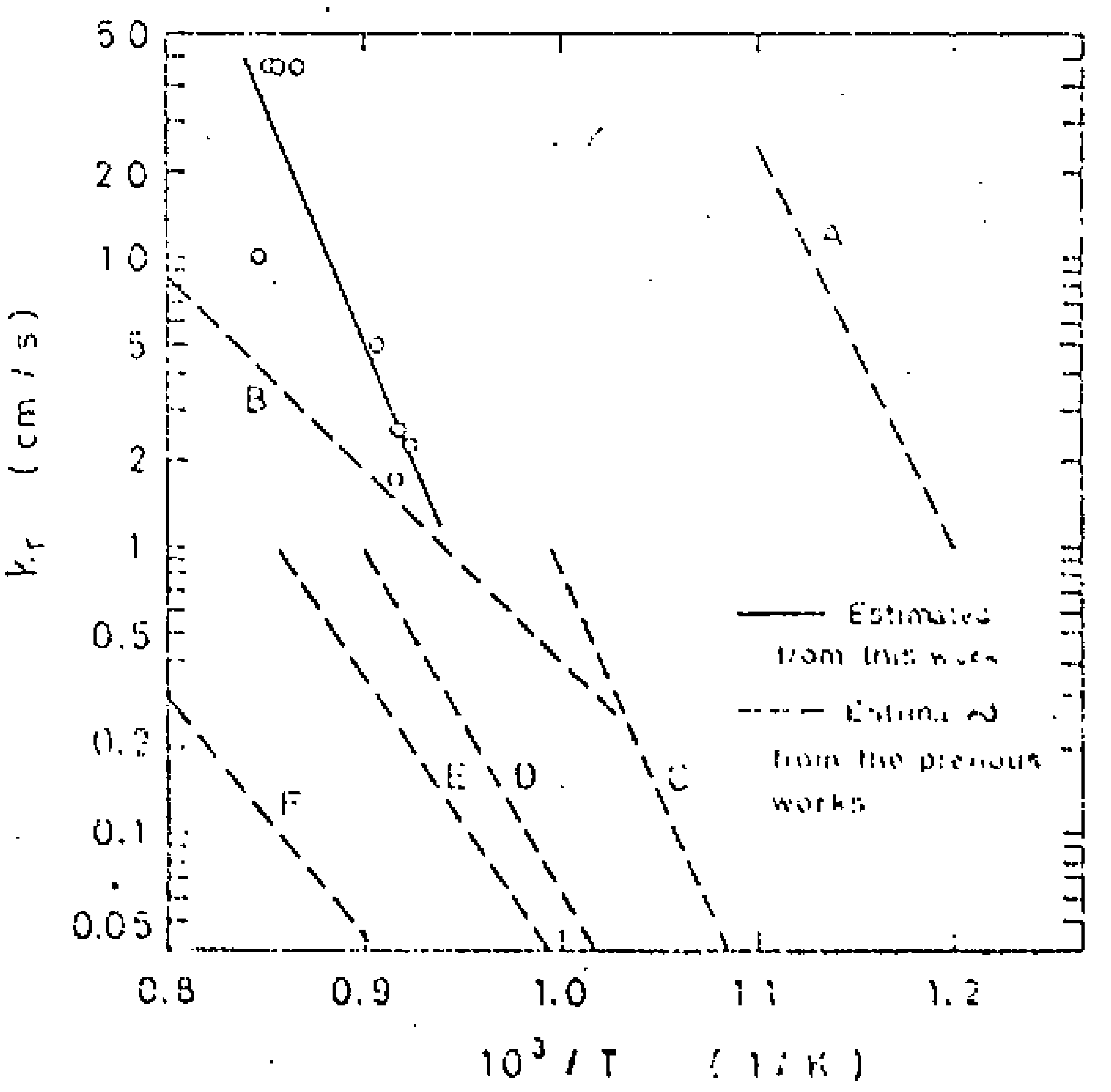

Fig. 4—Arrhenius plot of k_r. Broken lines *A* and *B*: Denbigh and Beveridge,[6] *C*: Gerlach and Stichel,[7] *D*: Cannon and Denbigh,[2] *E*: Ong, Wadsworth and Fassel,[1] *F*: Mendoza, Cunningham and Ronco.[7]

Table III. Experimental Conditions and Numerical Values of Parameters

Temperature, °C	v_0(cm³/s)	W_{ZnS}(g)	u_{mf}(cm/s)	u_0/u_{mf}(−)	L_f(cm)	δ(−)	d_B(cm)	K_{BE}(1/s)	k_g^B(cm/s)	k_g^E(cm/s)	D_e(cm²/s)	k_r(cm/s)
882	290	0.50	5.2	9.82	5.8	0.37	3.3	3.37	850	4.2	0.318	45
830	290	1.54	5.1	9.80	5.8	0.37	3.3	3.37	794	4.2	0.148	5.0
895	150	0.50	5.2	5.20	5.1	0.20	1.5	7.28	865	4.2	0.360	45
809	150	1.50	5.0	5.04	5.1	0.19	1.5	7.53	771	4.2	0.078	2.2
899	80	0.53	5.2	2.78	4.7	0.10	0.74	14.9	869	4.2	0.374	45
816	80	1.00	5.0	2.60	4.7	0.10	0.73	15.1	779	4.2	0.100	2.4
910	50	0.53	5.2	1.82	4.5	0.031	0.47	24.2	882	4.2	0.410	10
817	50	1.45	5.0	1.72	4.5	0.023	0.44	28.1	780	4.2	0.104	1.6

seen in this figure that the k_r values are within the variation of reported kinetic data, although our k_r values were estimated from an analysis of the fluidized bed with its complex hydrodynamics. The following Arrhenius equation was obtained by the least squares method:

$$k_r = 2.96 \times 10^{15} \exp\left(-\frac{75{,}005}{RT}\right). \qquad [32]$$

The activation energy of 75 kcal/mol is in fairly good agreement with 62 kcal/mol obtained by Denbigh and Beveridge,[6] 72 kcal/mol of Gerlach and Stichel[9] and 53 kcal/mol of Cannon and Denbigh.[5]

The O_2 gas concentration profile in the vertical direction was calculated at a fractional reaction of 0.5, and is shown by the solid lines in Fig. 5. It was presumed that the concentration profile of O_2 gas is affected by the d_B and K_{BE} values which were calculated from Eqs. [27] and [28], respectively. Eq. [27] proposed by Kobayashi[16] was obtained by correlating the data obtained by many workers[28-31] under different experimental conditions, and the maximum error in estimating d_B from this equation is about 30 pct on a logarithmic scale. The calculation of the concentration profile of O_2 gas was repeated by employing values of $2\,d_B$ and $0.5\,d_B$ as the bubble diameter, respectively. The results are also shown in Fig. 5 by dashed and dotted lines, respectively. Although the concentration profile of O_2 gas varies with the value of the bubble diameter, as seen in this figure, it can be said that the concentration difference of O_2 gas between gas bubbles and the emulsion phase depends on the flow rate of the fluidizing air: under a moderate bubbling condition at the flow rate of 80 cm³/s, the concentration difference is minor, and on the contrary, it is evident under vigorous bubbling conditions. This may suggest that it is preferable to employ the two-phase model in the analysis of gas-solid reactions conducted in a fluidized bed at higher flow rates of fluidizing gas and to separately evaluate the gas concentration in the gas bubbles and in the emulsion phase.

It may be interesting to further pursue the role of rising gas bubbles during the course of gas-solid reactions. The ratio of the amounts of ZnS particles reacted in unit time interval in gas bubbles and in the emulsion phase, ξ, was calculated by the following equation:

$$\xi = \frac{\int_0^{L_f} 4\pi r_o^2\left(\frac{N^B}{L_f}\right) k^B C^B\,dz}{\int_0^{L_f} 4\pi r_o^2\left(\frac{N^E}{L_f}\right) k^E C^E\,dz} = \frac{\delta\gamma}{(1-\delta)(1-\epsilon_{mf})}\,\frac{k^B\overline{C}^B}{k^E\overline{C}^E}. \qquad [33]$$

In this equation, the overall rate constants in gas bubbles and in the emulsion phase, k^B and k^E, are given from Eq. [4] as

$$\frac{1}{k^B} = \frac{1}{k_g^B} + \frac{r_o^2}{D_e r_i}\left(1-\frac{r_i}{r_o}\right) + \frac{1}{k_r}\left(\frac{r_o}{r_i}\right)^2 \qquad [34]$$

$$\frac{1}{k^E} = \frac{1}{k_g^E} + \frac{r_o^2}{D_e r_i}\left(1-\frac{r_i}{r_o}\right) + \frac{1}{k_r}\left(\frac{r_o}{r_i}\right)^2 \qquad [35]$$

respectively. The calculated ξ value was lower than 0.05 under the experimental conditions employed in this work. The low value of ξ was expected from the low γ value of 0.005, and it may indicate that the oxidation of ZnS particles in the gas bubbles is virtually negligible and the rising gas bubbles merely act as suppliers of oxygen to the emulsion phase.

Then, the fractional resistances of each step of gas-film mass transfer, pore diffusion and interfacial reaction in the overall reaction occurring in the emulsion phase, η_1, η_2, and η_3, were calculated by using

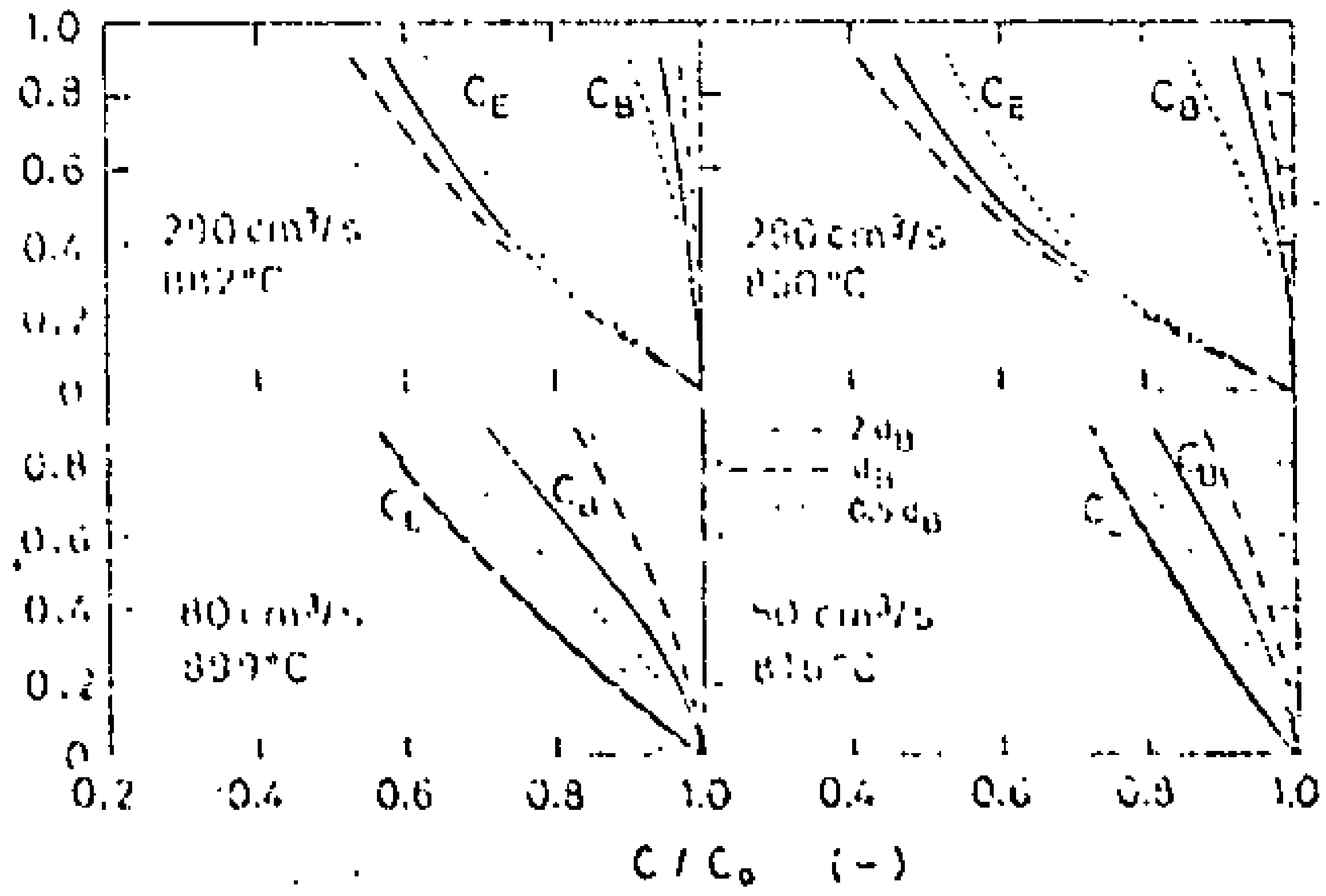

Fig. 5—Concentration profiles of oxygen gas in bubbles and in emulsion phase in vertical direction.

their respective terms on the right-hand side of Eq. [35]. They are:

$$\left.\begin{aligned} \eta_1 &= \frac{k^E}{k_g^E}, & \eta_2 &= \frac{k^E r_o^2}{D_e r_i}\left(1 - \frac{r_i}{r_o}\right) \\ \eta_3 &= \frac{k^E}{k_r}\left(\frac{r_o}{r_i}\right)^2. \end{aligned}\right\} \qquad [36]$$

The results of the calculation are listed in Table IV. It is seen in this table that the resistance of gas-film mass transfer is predominant at higher temperature and that the resistance of interfacial reaction also becomes conspicuous at lower temperature. The effect of the flow rate of air is not significant. This is expected from the assumption that the flow rate of gas in the emulsion phase is virtually maintained at the superficial velocity at incipient fluidization.

SUMMARY

The oxidation of ZnS particles in a batch-type fluidized bed was studied. In order to minimize the temperature rise of the fluidized bed during the course of reaction, less than 1.0 wt pct of ZnS particles were mixed with fused alumina particles, and the mixed particles were fluidized. Air was blown into the fluidized bed at flow rates of about 1.5 to 10.0 times u_{mf}. The temperature of the fluidized bed was chosen between 800 and 910°C. The SO_2 concentration in the exit gas was continuously measured by using an infrared gas analyzer, and it was corrected for the path volume between the fluidized bed and the gas analyzer. The fractional reaction was calculated from the corrected SO_2 concentration. The overall rate of reaction was increased by raising the flow rate of air and the temperature of the fluidized bed. The maximum temperature rise of the fluidized bed was less than 5°C, and the reaction was presumed to be isothermal.

The two-phase model was employed for the fluidized bed, and the rate equation concerning a single ZnS particle was derived on the basis of the unreacted core model. The reaction occurring at the interface ZnS/ZnO was assumed to be an irreversible first-order reaction with respect to the partial pressure of O_2 gas. The mass balance equations for O_2 gas in gas bubbles and in the emulsion phase were presented, respectively, and analytical solutions of the linear simultaneous differential equations were obtained.

The gas-film mass transfer coefficients on the surface of ZnS particles in gas bubbles and in the emulsion phase were estimated from the Ranz-Marshall equation and from previous work, respectively. The effective diffusivity of O_2 gas through the outer oxide shell was estimated from the measurement of pore size distribution by using the Wakao-Smith equation. The gas exchange coefficient between gas bubbles and the emulsion phase was calculated from the empirical equation proposed by Kobayashi. The rate constant of interfacial reaction was determined by the trial and error method so as to obtain a close fit between the theoretically calculated values of fractional reaction and the experimentally measured ones. The rate constants obtained and the activation energy of 75 kcal/mol were in fairly good agreement with those of previous workers.

Based on the mathematical model, concentration profiles of O_2 gas in the vertical direction in gas bubbles and in the emulsion phase were calculated. It was found that the concentration difference between both phases is trivial under the experimental condition of moderate bubbling. On the contrary, under the condition of vigorous bubbling, the decrease of O_2 concentration in gas bubbles is minor, which results in a significant difference in O_2 concentration between both phases. This is caused by the much higher upward flow rate of air in the form of gas bubbles than the rate of gas exchange.

The ratio of the amounts of ZnS particles reacted in unit time interval in gas bubbles and in the emulsion phase was found to be low under the experimental conditions, and the reaction mainly proceeds in the emulsion phase. The fractional resistances of gas-film mass transfer, pore diffusion and interfacial reaction in the emulsion phase were calculated. It was found that the resistance of mass transfer in the gas-film is predominant at higher temperature, and the resistances of both gas-film mass transfer and interfacial reaction contribute to the overall rate of reaction at lower temperature.

Table IV. Fractional Resistances of Each Step of Mass Transfer and Interfacial Reaction

	v_0 = 290 cm³/s, T_f = 862°C			v_0 = 290 cm³/s, T_f = 810°C		
	η_1	η_2	η_3	η_1	η_2	η_3
x = 0.1	0.904	0.005	0.091	0.519	0.006	0.475
0.5	0.843	0.029	0.128	0.415	0.029	0.556
0.9	0.611	0.097	0.292	0.186	0.060	0.754
	v_0 = 80 cm³/s, T_f = 899°C			v_0 = 80 cm³/s, T_f = 816°C		
	η_1	η_2	η_3	η_1	η_2	η_3
x = 0.1	0.905	0.004	0.091	0.346	0.005	0.649
0.5	0.850	0.024	0.126	0.253	0.028	0.719
0.9	0.622	0.083	0.295	0.103	0.048	0.849

NOTATIONS

C:	O_2 concentration,	(mol/cm³)
C_a:	SO_2 concentration measured by gas analyzer,	(mol/cm³)
C_f:	SO_2 concentration in exit gas from fluidized bed,	(mol/cm³)
C_i:	O_2 concentration at the reaction interface,	(mol/cm³)
C_s:	O_2 concentration at particle surface,	(mol/cm³)
d_B:	mean bubble diameter at the middle height of fluidized bed,	(cm)
d_p:	particle diameter,	(cm)
D:	diffusivity of O_2 gas,	(cm²/s)
D_e:	effective diffusivity of O_2 gas in ZnO shell,	(cm²/s)
k:	overall rate constant,	(cm/s)
k_g:	gas film mass transfer coefficient,	(cm/s)
k_r:	rate constant of interfacial reaction,	(cm/s)
K_{BE}:	gas exchange coefficient between gas bubbles and emulsion phase,	(1/s)

L_f:	height of fluidized bed,	(cm)
L_{mf}:	height of fluidized bed at incipient fluidization,	(cm)
M:	molecular weight,	(g/mol)
n:	mole number,	(mol)
N:	number of ZnS particles,	(—)
r_i:	radius of reaction interface,	(cm)
r_o:	radius of particles,	(cm)
S:	cross sectional area of fluid bed,	(cm^2)
(pct S):	percentage of sulfur in ZnS particles,	(—)
t:	time,	(s)
T:	absolute temperature,	(K)
u_B:	rising velocity of gas bubbles,	(cm/s)
u_o:	superficial velocity of fluidizing gas,	(cm/s)
u_{mf}:	incipient fluidization velocity,	(cm/s)
u_t:	terminal velocity,	(cm/s)
v:	flow rate of gas,	(cm^3/s)
v_o:	flow rate of fluidizing gas,	(cm^3/s)
V:	volume of backmix flow,	(cm^3)
W_{ZnS}:	weight of ZnS particles fed to the reactor,	(g)
x:	fractional reaction,	(—).
z:	height from the bottom of fluidized bed.	(cm)

Greek Letters

γ:	volume fraction of fluidized particles in gas bubbles,	(—)
δ:	volume fraction of gas bubbles in fluidized bed,	(—)
ϵ:	porosity, void fraction,	(—)
ϵ_{mf}:	void fraction at incipient fluidization,	(—)
θ:	mean residence time,	(s)
μ:	viscosity,	(g/cm · s)
ν:	kinematic viscosity,	(cm^2/s)
ξ:	ratio of the amounts of ZnS particles reacted in gas bubbles and in emulsion phase,	(—)
ρ_g:	density of gas,	(g/cm^3)
ρ_p:	density of fluidized particles,	(g/cm^3)
ϕ:	fraction of ZnS particles in the total fluidized particles.	(—)

Superscripts

B:	gas bubbles,
E:	emulsion phase.

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APPENDIX D:

Cominco, Trail Operations:

Description of main production facilities

Trail Operations

A basic technical description
of the main production
facilities

Cominco

Trail Operations of Cominco

Foreword

The following material has been prepared to provide a basic technical description of the main production operations of Cominco at Trail, British Columbia. It does not discuss environmental control related to these operations nor does it reflect other activity of the company's B.C. Group at this location. The content is devoted to:

Introduction

Cominco Ltd. is one of Canada's major companies (97% of its shares are held in Canada) and has widespread operations and interests in Canada and other countries. It originated at Trail, B.C. in 1906 as a consolidation of a smelter and lead-silver refinery owned by the Canadian Pacific Railway with some mining interests in the area.

In 1909 the company acquired the Sullivan Mine, at Kimberley, B.C., 200 rail miles east. Selectively mined lead-silver values were shipped to Trail over the next decade and lead smelting and refining expanded.

In 1916 the company purchased the common stock of the West Kootenay Power and Light Company, which brought hydroelectric developments and potential near Trail into the corporate structure. Also in 1916 the company began zinc production on a small scale. In 1920 the development of a differential flotation process made possible the separation of the complex run-of-mine Sullivan ore into a high grade lead concentrate and a high grade zinc concentrate. These developments were the foundation for the rapid growth which followed at Trail.

In 1931, to remove the harmful effect of sulphur dioxide emissions on vegetation, particularly across the international border 10 miles south, the company began producing sulphuric acid for utilization in the manufacture of chemical fertilizers.

Although there was some extension of the company's interests beyond the immediate Trail-Kimberley area before World War II, this aspect of corporate development began to have greater impetus starting in the 1950's. It is now far-reaching in scope and diversity.

Trail continues to be the largest production centre in the company's structure. Trail is also headquarters for the company's B.C. Group, which, in addition to serving B.C. operations, serves other company developments as required.

Engineering is the largest of these. This organization includes extensive shops, technical service facilities such as for materials engineering and instrumentation, and planning and design staffs who deal with various corporate projects, often of substantial scope. The technical research centre also has broad capabilities. Other core functions have responsibilities for mine development and engineering, environmental control, personnel, labour relations, purchasing, and other aspects of administration. The corporate computer centre is also located at Trail.

The company employs about 3,800 at Trail, of whom approximately 2,500 are employed within Trail Operations.

April, 1977

Zinc Operations

To meet the demand of the Allies for zinc in the first world war, Cominco pioneered a process to use the readily available supply of zinc sulphide from the Sullivan Mine and hydroelectric power from the Kootenay river. The operation that started in 1916 resulted in overall production over the next three years of 20,000 tons of electrolytic slab zinc.

Because of the complexity of the Sullivan ore, economic separation into a clean lead and zinc concentrate was not possible. To achieve high recoveries of both metals the advantages of a closely integrated lead smelting - zinc production operation was recognized from the outset. This objective was achieved in 1930 when the slag fuming and oxide leaching plants went into operation. High recovery of zinc, lead and silver from both concentrates was then realized. This development plus a number of plant expansions in the next three decades brought production up to nearly 600 tons per day by 1960.

Recent developments have seen the expansion of the roasting plant by adding a 350 ton per day suspension roaster in 1961 and the replacement of the eight - 150 tons per day suspension roasters by two 650 metric tons per day Lurgi turbulent layer roasters in 1971.

At present a 40 ton per day electrolytic pilot plant incorporating jumbo size cathodes, external cooling and automatic stripping is operating as a forerunner to the modernization of the existing electrolytic and melting facilities. A new acid plant replaces two obsolete units and allows more effective treatment of smelter and roaster gases.

Capacity is now about 750 tons per day of refined zinc and 4000 pounds per day of cadmium. In addition 500,000 oz. per year of indium are produced in facilities administered by Zinc Operations. Some 15% of refined zinc production is supplied from the slag fuming plant. The fume is the end product from zinc plant residues and zinc in lead concentrate.

Sulphur recovery plants process roaster gases and ammonium bisulphite from the smelter to recover the following:

Sulphuric Acid	1000 tons per day
Ammonium Sulphate in Solution	330 tons per day
Liquid SO_2	160 tons per day
Ammonium Bisulphite	15 tons per day

These products are either sold directly or shipped to the fertilizer plants for phosphate or sulphate fertilizer production.

Zinc Operations are divided for administration into five sections or plants. Each of these is directed by a plant superintendent, an operating foreman and shift bosses. In general, maintenance is provided by crews assigned to each plant, who are directed by plant maintenance foremen. Plant maintenance groups are supported by a central group who assist in planning, scheduling, processing and in the training of crews. The maintenance organization is administered by a superintendent and a general maintenance foreman.

Overall responsibility rests with the superintendent of zinc operations and an assistant. The approximate labour usage broken down by plants is as follows:

Zinc Labour on Roll

	Staff	General Roll
Overhead and Clerical	15	15
Roasters & Sulphur Recovery	15	110
Sulphide Leaching	8	60
Oxide Leaching, Cadmium & Indium	8	90
Electrolytic	13	300
Melting and Casting	6	150
Assigned Maintenance	14	110
	80	835

Roasting and Sulphur Recovery

Roaster feed is predominantly Pine Point and Sullivan Concentrate. The remainder, some 10%, is from Cominco's H.B. Mine and customs shippers.

Over 90% of the concentrate is received in bottom dump hopper cars. The remainder is received by truck. To provide flexibility in roaster operation each roaster is provided with two feed bins. This is especially important in controlling the lead content of the feed to the turbulent layer roasters. Typical analyses of major concentrate sources are as follows:

	Zinc	Lead	Iron
Sullivan	49%	6%	11%
Pine Point	58%	1%	4%

The Lurgi roasters, sized at 84 sq. meters bed area, operate continually except for a 10-day annual overhaul on each. The suspension roaster is operated on a part-time basis depending on calcine inventory. With the fluid bed roasters about 50% of the calcine product is from the bed overflow and is cooled in fluid bed coolers. The remaining calcine is removed from boilers and cyclones, with less than 5% being collected in the electrostatic precipitators. Calcine is ground by ball mill, air classified and pneumatically conveyed by a Fuller Kenyon system to two 2,000 ton storage silos at the leaching plant. Gases from the electrostatic precipitators of the Lurgi roasters and the glass fiber bag house of the suspension roaster are combined, cooled and scrubbed in packed towers, and transported by lead-lined flue to the Sulphur Recovery Plant.

Gas volume at this stage is about 100,000 cfm at about 8.5% SO_2. The bulk of the gas goes directly to three single stage contact acid plants. The acid plants are all Monsanto four pass design ranging in size from 450 tons per day to 900 tons per day. Tail gas from the acid plants passes through ammonia scrubbing towers and a Brink mist eliminator prior to discharge to atmosphere. Minor amounts of the roaster gas are treated in an ammonia absorption plant to produce ammonium bisulphite. The bisulphite from the zinc and lead plants is then combined and treated with H_2SO_4 to produce SO_2 and ammonium sulphate. The 100 per cent SO_2 gas can be sent to the acid plants or compressed and condensed for sale as liquid SO_2. The ammonium sulphate is pumped to the Warfield Fertilizer Plant as a 40% solution.

Calcine Leaching

The calcine leach operation employs air agitated pachucas. The process is a continuous two-stage leach. In the first stage acid leach, 80-90 percent of the zinc calcine is mixed with cell room return acid and leached to a pH of 2.5 in seven acid pachucas.

Leaching sands, separated by a cone classifier, are ground in a ball mill and recycled to the acid pachuca leach.

After thickener separation the leach residue is filtered and washed through Moore basket filters and two stages of American disc filters. Leach residue containing 35 percent moisture is pumped to the lead smelter for subsequent lead and zinc recovery.

Acid thickener overflow solution is neutralized with calcine and agitated through two parallel circuits of six pachucas each. After neutral thickening the thickener underflow solids are recycled to the acid leach pachucas.

Before zinc dust purification the clear neutral thickener overflow at 50°C is heated to 72°C using a Croll Reynolds vacuum heat exchanger.

Solution flow is continuous through two stages of zinc dust purification. The first stage consists of five mix tanks in series and the second stage consists of two mix tanks. Fresh zinc dust at a rate of 30 pounds per ton of cathode is added to the second stage of purification. The zinc dust is separated by a cyclone and passed to the first stage of purification. Filter presses are used after the first and second stages.

Solution analysis before and after purification is as follows:

	Zn gpl	Cd mg/l	Cu mg/l	Co mg/l	Ge mg/l	Sb mg/l	Ni mg/l
Before	143	200	50	1.0	0.1	0.08	0.5
After	144	0.2	<0.1	0.2	0.01	0.02	<0.1

Final cooling of the purified zinc sulphate solution to 25°C is done in a forced draft atmospheric cooling tower. Precipitated gypsum is removed in a clarifying thickener.

Oxide Fume Leaching

Zinc oxide fume from the lead smelter slag fuming furnaces is leached in a separate leaching plant. In addition zinc dust purification residues from the calcine leaching plant are dissolved in acid and added to the oxide leaching acid pachucas. Leaching of the fume is done in an acid stage followed by a neutral stage using pachucas. Because of the very high impurity content of the oxide fume approximately 2.5 gpl of ferric iron is added to the acid leach pachucas. Precipitation of ferric hydroxide in the neutral pachucas co-precipitates a major percentage of germanium, arsenic, and antimony from solution. The iron additive solution is produced by leaching calcine leach residue with concentrated sulphuric acid at 90°C.

After settling in neutral thickeners the thickener overflow is pressure filtered (Kelly filter) before zinc dust purification. The neutral thickener underflow is acid leached at pH 4.0 and then filtered and washed by Kelly filters followed by two stages of disc filtration.

Solution purification with zinc dust is done in three stages at 75°C. The first stage produces a high cadmium residue which forms the primary feed for cadmium production plant.

Electrowinning

The tank room consists of 2160 cells arranged in units of 36 cells with four cascade rows of nine cells each. Each cell contains 22 aluminum cathodes and 23 lead (0.75% silver) cast anodes. The cells are constructed of concrete lined with fiberglass. The current density employed is in the range of 600 to 800 amperes per sq. meter and the current efficiency is about 90 percent. Neutral purified zinc sulphate solution is metered to each cell to maintain a zinc concentration of 55 gpl and a free acid content of 145 gpl. Heat generated by the electrowinning is removed by two lead pipe cooling coils located at the end of each cell. Operating cell temperature varies from 34°C to 40°C depending on the condition of the cell and the cooling water temperature.

Heat generation increases as a layer of MnO_2 builds up on anode surface. Units are removed from production every 42 days to remove excess MnO_2 from the anodes and to remove accumulated material from the bottom of each cell. The cathodes are normally stripped every 24 hours but a shorter plating time of 16 hours may be used on units operating at high current density. Stripping of the zinc deposits is done manually.

At the present time a large 40-ton per day pilot group is operating to demonstrate a design for modernization of the electrolytic plant. This pilot group features large sized anodes and cathodes, mechanical stripping of zinc, low current density operation (400 amps/m^2) with a 48 to 72 hour plating cycle, and external cooling tower heat removal.

Melting and Casting

The cathode is melted in low frequency induction furnaces. The original installation was about 20 years ago. At that time the plant was rebuilt and three SICE furnaces were installed. To meet expanding production a 3000 K.W. Ajax type furnace was added in 1971.

A full range of product analyses and shapes is made. These include special high grade, high grade, prime western and a number of alloys containing minor amounts of lead, aluminum, cadmium, copper and iron.

About 65% of this production is cast in 56 pound slabs using three Sheppard casting machines. The remainder is cast in 2400 pound jumbos of three basic shapes. One of these shapes is Cominco's C-Cast jumbo which is made by sawing sections from 15 foot semi-continuous cast logs. This product is cavity free, saves 23% on storage space and has a weight tolerance of ± 1%.

A typical assay of special high grade zinc is:

Lead - 0.0017%max.
Cadmium - 0.001% max.
Copper - 0.001% max.
Iron - 0.002% max.
Zinc - 99.99% min.

Cadmium Production

Cadmium production is closely integrated with the zinc oxide fume leaching plant. First stage oxide plant purification residue is leached to dissolve zinc with dilute sulphuric acid to remove excess zinc. After filtration the residue is further leached in H_2SO_4 to dissolve cadmium. This solution is filtered and then treated with $KMnO_4$ and lime to remove thallium and other impurities. After filtering, the purified solution is cemented with zinc dust to produce cadmium sponge which is filtered, briquetted and melted. The cadmium metal is cast into balls or extruded in rods for sale to the cadmium plating industry.

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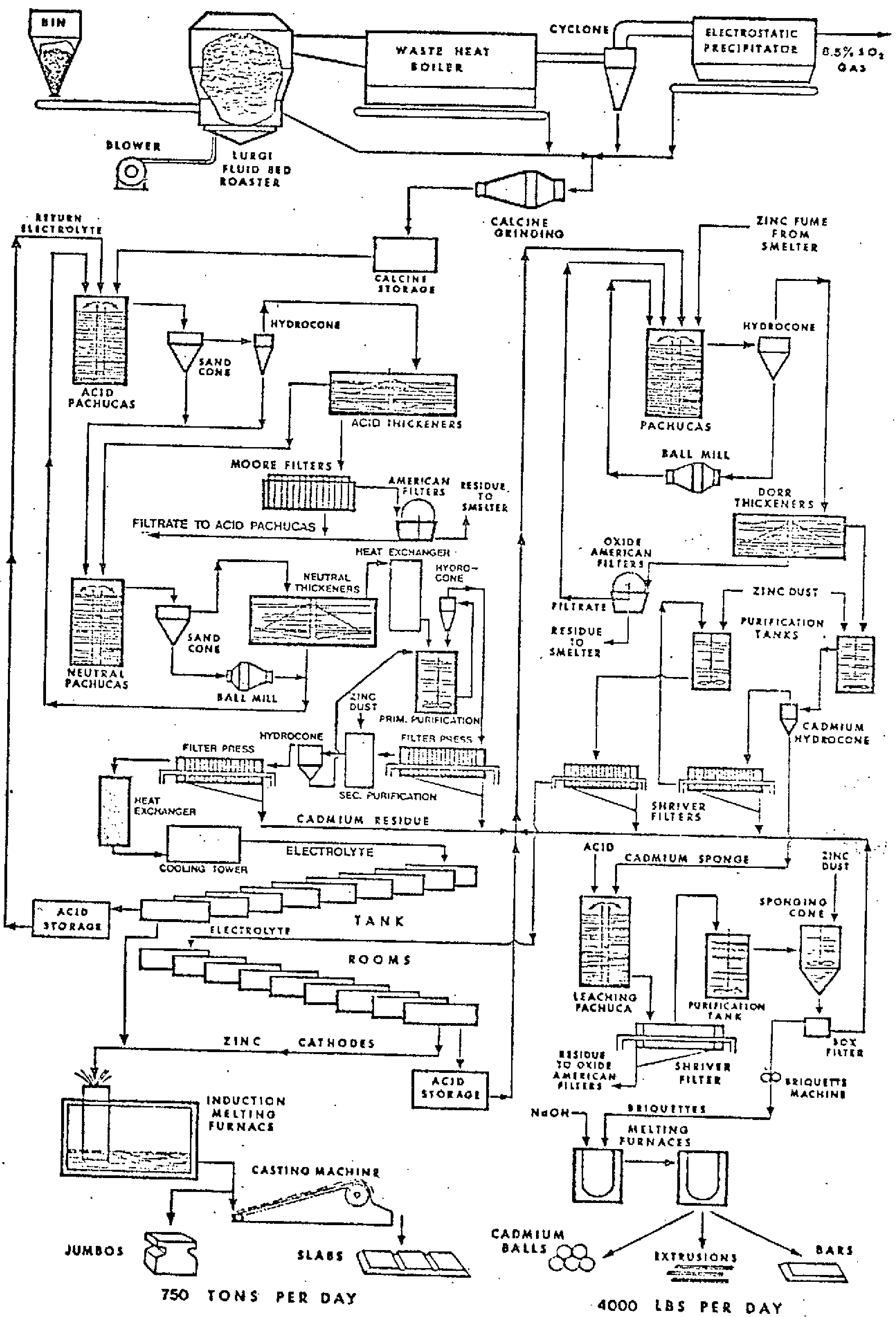
SIMPLIFIED FLOWSHEET
ZINC OPERATIONS



Lead Smelting

The Lead Smelter is located on the site of the original Trail Smelter built by F. Augustus Heinze in 1896 to smelt the copper-gold ores from the Rossland mines.

Smelting of lead ores began in 1899. Through the years the smelting of copper ores diminished as ore supplies were depleted. Lead smelting, particularly after the acquisition of the Sullivan Mine, expanded. Many technical changes have been made through the years. A major innovation was the recovery of zinc from the smelter by slag fuming which began in 1930, with a second fuming furnace added in 1947. The present sintering plant was commissioned in 1953 while the original brick shaft furnaces were replaced with water jacketed shaft furnaces in the 1940's.

The smelter produces 500 tons per day of lead bullion in the form of anodes for the lead refinery, 210 tons a day of zinc oxide fume for treatment in zinc operations and 10 - 15 tons a day of copper matte, some of which is roasted by zinc operations for copper sulphate production and the remainder sold to European smelters.

Sinter machine off-gas provides 320 tons a day of sulphur dioxide. This gas is absorbed in an ammonium bisulphite solution and subsequent acidification of this product solution produces pure sulphur dioxide for liquid sulphur dioxide sales and ammonium sulphate fertilizer product.

Lead concentrates from Cominco's Sullivan, Pine Point and H.B. mines, and custom concentrates from other sources are treated together with residues from the Zinc Operations oxide and sulphide leaching plants. Silica and limerock flux are purchased locally while coke, coke breeze, and coal are purchased from Kaiser Resources at Sparwood, B.C. Refinery by-products and smelter recycle materials make up the remainder of the feed.

Lead smelting is divided into two main areas, sintering and furnaces. The smelter has its own maintenance service group as well. A general superintendent with one assistant superintendent administer the operation. Plant superintendents are responsible for the sinter plants, furnaces and maintenance services. Foremen and shift bosses have direct responsibility for their crews.

A breakdown of manpower is shown below:

TABLE 1

SMELTER		MANPOWER
	Staff	General Roll
General	8	---
Sinter Plt.	18	131
Furnaces	20	174
Maintenance	15	79

Sintering

Most concentrates are received in bottom dump railway gondola cars at three unloading bays. Cars are pipe sampled for assaying after weighing. Numerous concentrates are received (at least 24 in 1974); the major sources are stocked in bins while others are mixed together in a bedding plant. Flux shipments by truck are received at the bedding plant. Concentrates and fluxes are transferred to the dry feed plant bins by front end loader. In the dry feed plant, the materials are proportioned according to the charge calculation, using 12 feed bins of 80 - ton capacity with adjustable gates and variable speed belts. A common collector belt transfers the feed to the sinter plant. The sinter charge is carefully prepared by the plant metallurgist with computer assistance to provide a suitable blend of materials to yield a low sulphur sinter as well as suitable slag and bullion composition at the blast furnaces.

The dry feed is mixed with return sinter and zinc plant residue slurry in three 80' natural gas-fired rotary dryers to nodulize and dry the mixture into suitable sinter machine feed. A drum nodulizer is used for final nodule control to ensure that nodules are well formed, properly sized (less than 1/2") and contain at least 5.5% water. Because the residues have little fuel value, coke breeze is added to obtain proper fusion on the sinter machines.

The down-draft sinter machines (10' x 107') in parallel lines reduce sulphur from 7% to 2%, normal production is 1,600 tons per day of sinter. The sinter is passed over grizzlies to remove fines. The fines plus some coarse sinter are recycled to two roll crushers providing a circulating load of return sinter necessary to dilute the sulphur content of feed and to provide nuclei for nodules. The recycle material represents about 50% of the feed to the sinter machines.

Lead on charge is maintained at 36% - 40%. Metallic lead plugging sinter machine grates and interfacing with pallet movement becomes a serious problem above 42% lead with the down-draft sinter machines. Any metallic lead dripping into the wind box, is granulated in water. The off-gas from sintering, 1.5 - 2.0% SO_2, is humidified, cleaned by electrostatic precipitation and scrubbed with ammonium hydroxide - ammonium bisulphite mixture to reduce sulphur dioxide to 0.1% SO_2. The cleaned gas is then discharged to atmosphere through a 400' stack.

Furnaces

Sinter and coke, in weighed proportions of 10 to 1, are charged to the blast furnaces using a mono-rail bucket tramway. Two water-jacketed blast furnaces produce 500 tons a day of bullion and about 625 tons a day of slag. The blast furnaces are 6' x 24' and 18' high. Blast air is enriched to about 24% oxygen. Typical assays of Smelter products are shown in Table II.

Gas from the blast furnaces is cooled and cleaned through a 20 - compartment Wheelabrator baghouse (108,240 sq. ft. of cloth) and the tapping area of the furnaces is ventilated by a 6 compartment Wheelabrator baghouse (39,072 sq. ft. of cloth). The cleaned gases from the baghouses are vented to atmosphere through a 180' stack. The Baghouse dusts are pneumatically pumped to the slag furnaces and are the major purge of cadmium from the smelter.

Bullion and slag are continuously tapped from the furnaces by means of Roy tappers into settlers from which the slag and bullion flow to transfer pots. Slag analysis is controlled through sinter plant charge adjustment to ma[illegible] 20.5%, CaO at 10.5% and iron around 26% to satisf[illegible] basicity ratio $\frac{(CaO + Fe)}{(SiO_2}$ of 1.8.

The bullion is transferred by crane to the drossing furnace where cooling causes copper dross formation. The dross is removed by hand raking. It is then reheated in a dross retreatment furnace from which copper matte, slag and bullion are tapped periodically. This bullion is returned to the drossing furnace. Drossed bullion is further purified in small floor pots to remove tin dross, then cast as anodes on a continuous casting wheel. The anodes, 98.4% lead, are trammed to the Refinery. Antimony, arsenic, bismuth and tin content are controlled by charge adjustments at the sinter plant and at the blast furnaces.

The blast furnace slag is batch-charged to two slag fuming furnaces from which zinc and lead oxide are fumed using coal and air. The fume-carrying gases pass through waste heat boilers and following, a Dracco baghouse (54,000 sq. ft. of cloth) for fume recovery. The fume is shipped in covered gondola railway cars to the zinc oxide leaching plant for recovery of zinc and cadmium.

Barren slag from the slag fuming furnaces is discarded.

TABLE II

TYPICAL ASSAYS OF SMELTER MATERIALS

	Zn	Pb	Fe	Cu	SiO_2	CaO	Bi	Sb	As
Blast Furnace Slag	18.5	4.0	25.0	.25	20.5	10.5	---	---	.10
Bullion	----	98.4	----	.07	----	----	.12	1.0	.35
Fuming Furnace Fume	53.0	21.5	.3	.10	.4	.1	---	---	.55
Fuming Furnace Slag	2.5	.10	31.0	.30	30.0	17.0	---	---	---
Copper Matte	.25	25	.4	52	----	----	---	---	3.0

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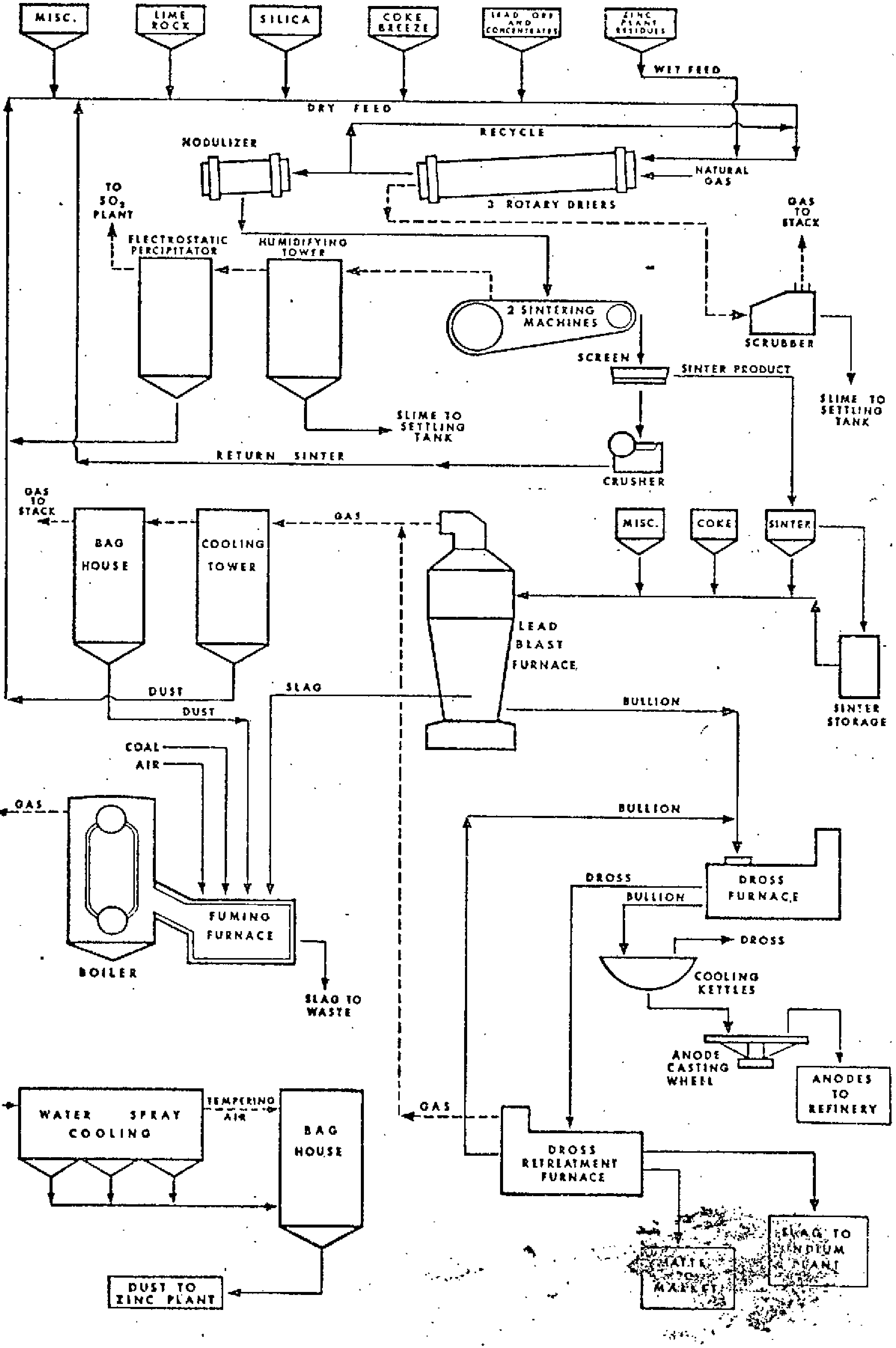
SIMPLIFIED FLOWSHEET
LEAD SMELTING



Refineries

The Lead Refinery at Trail, which began production in 1902, was the first commercial application of an electrolytic lead refining process - the process developed by Dr. Anson G. Betts in 1901. The original installation consisted of 240 asphalt - lined wooden cells with a rated capacity of 10 tons of lead per day. Expansion and process improvements over the years have brought the plant to 1004 cells and a capacity of about 500 tons of refined lead per day. Also additional processes have been added for the recovery of silver, gold, bismuth, antimony (in the form of antimonial - lead alloy) and arsenic. The Betts electrolytic process continues to be the only commercial refining process capable of producing high purity lead from the metallurgical complex concentrates handled at the Trail Operations.

Production of minor metals varies from year to year depending on values in concentrates processed in the smelter. Normal production is 240 thousand pounds per year of bismuth, 800 tons per year of antimony, 10 million ounces per year of silver and 10 thousand ounces per year of gold.

In 1974 vacuum distillation equipment was installed capable of recovering 100 tons per year of crude arsenic metal.

The refinery complex employs a total of 190 people including 25 supervisory and office staff and 24 in maintenance and repair work. In recent years development work has been concentrated in the areas of process technology and mechanization. Improvements have resulted in improved reliability of the processes, better working conditions of our employees and improved overall efficiency of production.

Lead Refinery

Impure lead bullion from the smelter is delivered to the refinery in the form of cast anodes. Each refining cell is set with 24 anodes and 25 pure lead cathodes. Pure lead is corroded from the anodes and deposited on refined lead cathode starter sheets. Electrolyte containing 95 g/l H_2SiF_6 and 70 g/l dissolved lead is circulated through the cells. Organic addition agents, lignin sulphonates and an extract of the aloes plant, are added to the electrolyte to assist in producing dense, level cathode deposits. Electrically the cell room is divided into the three independent circuits supplied with power from motor generator sets. The cathodes and anodes in each cell are connected in parallel while the cells in each circuit are series connected. Average current density over the seven day plating cycle is about 19 amps/sq.ft.

At the end of seven days the cathodes and anodes are removed and the cells reset with new anodes and cathodes. The cathode deposits are washed and then melted in a 235-ton capacity pot furnace. The molten lead is given a final flash treatment with caustic soda to remove traces of antimony, arsenic and tin and is then ready to be cast into ingots for sale.

The bullion impurities consisting of silver, gold, bismuth, antimony and arsenic are retained on the anodes in the form of a black slime. This slime is scraped from the anodes, washed and partially dried before being processed in the silver refinery for the contained metals. The lead remaining at the anode after seven days of electrolysis amounts to 30 to 35% of the original weight and is remelted and cast into new anodes.

Silver Refinery

The first step in the processing of slime for the recovery of minor metals in the melting of the slime to produce a "black metal" containing 2 to 8 oz/ton of gold, 3,000 to 7,000 oz/ton of silver, 20% lead, 30 to 40% antimony, 10 to 20% arsenic, 5 to 10% bismuth and 3 to 4% copper. A minor amount of slag containing lead, antimony and arsenic oxides is also formed during slime melting and is recycled back to the smelter blast furnaces.

The "black metal" is then transferred to a burn down furnace where antimony and arsenic are preferentially oxidized at 1400 to 1600°F. These oxides along with dusts from the slimes melting operation are recovered in the furnace ventilation bag houses and are processed for the recovery of antimony and arsenic.

The metal from the burn down operation is transferred to a "dore" furnace and heated to 1800 to 2000°F where air oxidation removes the lead, bismuth and copper in the form of litharge slag. Litharge slag is reduced in a reverbatory furnace to produce a 30% bismuth-lead alloy. This alloy, after drossing to remove copper and Parks process disilverizing, is treated with chlorine to produce pure bismuth, metal and by-product lead chloride.

The metal from the "dore" furnace, containing over 99% silver and a small amount of gold, is cast into thin slabs which form the anode in a Thrum-type electrorefining cell. Silver crystals formed on the cathode are separated from the silver nitrate electrolyte and melted into 1000 troy ounce bars. Gold is recovered from anode compartment black slimes.

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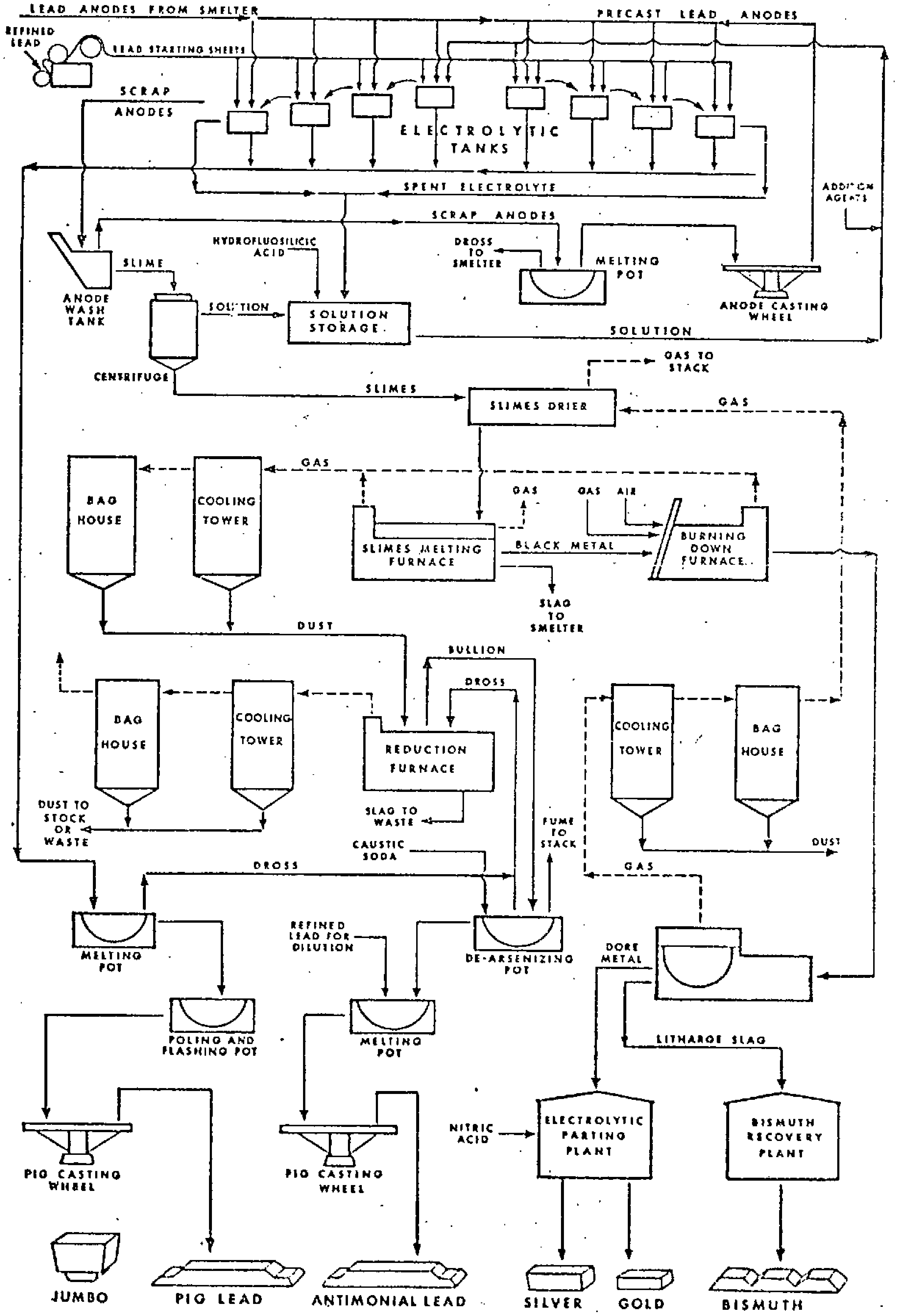
SIMPLIFIED FLOWSHEET
LEAD REFINERY



Fertilizer Operations (Warfield)

Since the plants began operating in 1930 a number of major changes have taken place. Fertilizer production expanded gradually in the 1930's with product acceptance. Construction was required in World War II to produce ordnance grade ammonium nitrate, and this was followed by expansion to meet the heavy post-war demand for fertilizers. In more recent years, with the company having established fertilizer production facilities at more strategic locations, fertilizer manufacture at Trail has been directed mainly to the objective of efficient control and utilization of metallurgical sulphur.

For convenience the plants comprising the fertilizer operations are referred to as the ammonia plants, comprising the natural gas ammonia plant, the air separation unit and the ammonia synthesis plant; the fertilizer plants, comprising the phosphate plant and the ammonium sulphate plant; and storage and shipping. The ancillary functions include maintenance, instrumentation, other technical and clerical work.

The operations employ about 300 people. Of these about 54 operate the ammonia plants, 68 operate the fertilizer plants, 27 are employed in shipping, 115 work out of the Warfield maintenance centre, instrument shop and yard office, and 36 perform clerical, technical and general work.

Natural Gas Ammonia Plant

The natural gas ammonia plant was built in 1940 to make ammonia synthesis gas from coke. In 1958 the plant was converted to natural gas, and in 1964 it was altered to its present form and capacity of 200 tons per day of ammonia.

The natural gas ammonia plant uses natural gas supplied by Inland Natural Gas from West Coast Transmission and the Peace River gas fields or alternating from Alberta Southern Natural Gas. The gas is desulphurized, reformed with steam and oxygen-enriched air, and treated to produce a gas mixture suitable for the synthesis of ammonia.

The basic reactions are:

a. Reforming:

$$\underset{\text{methane}}{CH_4} + \underset{\text{steam}}{H_2O} \xrightarrow[\text{nickel catalyst}]{\text{heat}} \underset{\text{carbon monoxide}}{CO} + \underset{\text{hydrogen}}{3H_2}$$

b. Shift:

$$\underset{\text{Carbon monoxide}}{CO} + \underset{\text{steam}}{H_2O} \longrightarrow \text{iron catalyst followed by zn copper catalyst} \quad \underset{250^\circ C}{400^\circ C} \longrightarrow \underset{\text{carbon dioxide}}{CO_2} + H_2$$

c. Methanation: (following removal of most of the CO_2 using Sulfinol scrubbing solution)

$$CO_2 + 4H_2 \longrightarrow 300^\circ C \longrightarrow CH_4 + 2H_2O$$

$$CO + 3H_2 \longrightarrow \underset{\text{catalyst}}{\text{nickel alumina}} \longrightarrow CH_4 + H_2O$$

d. Synthesis:

$$\underset{\text{hydrogen}}{3H_2} + \underset{\text{nitrogen}}{N_2} \longrightarrow \overset{4000\text{ psi}}{\underset{\text{iron catalyst}}{500^\circ C}} \longrightarrow \underset{\text{ammonia}}{2NH_3}$$

A natural gas flow of 3400 cfm enters the process at 200 psi. Sulphur compounds, which would poison catalysts, are removed to a content of less than 1 ppm, by absorption on activated carbon and by cobalt-moly zinc oxide at 400°C.

The primary reformer consists of 36 twenty-nine foot 6" i.d. HK-40 tubes filled with nickel catalyst. It is fired with 1000 cfm of gas. The steam to process gas ratio is about 3 to 1. Exit gas temperature is 650°C.

The secondary reformer contains 300 cubic feet of nickel catalyst. About 5000 cfm of air, enriched to about 30% oxygen, enters the burner at the top. Exit gas temperature is 900°C. Gases are cooled in a boiler generating 20,000 lb. per hour of steam, and by a condensate quench.

Then the gases pass through 827 cubic feet of high temperature shift catalyst at 400°C followed by 785 cubic feet of low temperature shift catalyst at 250°C. The gases are then cooled and compressed from 95 psig to 235 psig. CO_2 is then removed from the gas stream by absorption in Sulfinol. The gases are then heated and passed through 300 cubic feet of catalyst at 300°C in the methanator, where the CO and CO_2 still remaining are converted to methane. This synthesis gas containing basically a 3 to 1 mixture of hydrogen to nitrogen is now compressed in three stages to 4000 psig and piped to the synthesis plant.

Air Separation Unit

The air separation unit was installed in 1972 to supply oxygen to the smelter and the natural gas ammonia plant following shutdown of the electrolytic hydrogen plant and three small liquid air units.

Air is compressed to 600 psig in the first four stages of three 6-stage compressors. These machines are balanced with nitrogen in the 5th and 6th stages. The compressed air is purified by a caustic wash, and dried by cooling to -5°C followed by moisture absorption on alumina, to provide a dew point of less than -60°C. The air separation plant is an Air Liquide unit. With the available air compression capacity the unit produces 125 tons per day of 98.6% oxygen. Nitrogen is produced at 99.9% purity. About 40 tons per day of oxygen is used by the natural gas ammonia plant, and the remainder is used by the smelter.

Ammonia Synthesis Plant

The ammonia synthesis plant was built in 1930 to produce ammonia from electrolytic hydrogen and liquid air nitrogen. There are five synthesis columns, with the auxiliary circulators and condensers although only four are presently in operation. If enough synthesis gas were available the five units could produce 400 tpd of ammonia.

The four active synthesis columns are of the Chemical Construction Corporation design, each with internal heat exchange by tube bundles in the catalyst mass, which weighs close to 5 tons with a volume of about 70 cubic feet. Circulation capacity in each of these four older columns is approximately 15,000 cfm. Make-up synthesis gas enters at the ammonia condenser where water and CO_2 residues are absorbed into the liquid ammonia. Oxygen-containing compounds are catalyst poisons. Circulating gas enters the columns containing 2% ammonia and leaves containing 12 1/2%. A small part of the circulating gases is purged to keep the inert gases, methane and argon, below about 18%. The ammonia is let down in pressure from 4,000 psi to 175 psi allowing dissolved gases to be released. This involuntary purge gas, along with other purge gases from condensers, contains ammonia and is scrubbed with condensate. This ammonia water goes to the aqua plant where liquid ammonia is added to make a 38% aqua ammonia solution used for sulphur dioxide absorption from metallurgical gases.

Liquid ammonia is stored in three spheres totalling 1790 tons capacity. Eight small compressors can liquify about 150 tons per day of 12 psig ammonia gas. Liquid ammonia can be loaded or unloaded from rail tank cars, but the maximum production rate essentially balances that required by the metallurgical plants for the capture of sulphur.

Phosphate Plant

The Warfield phosphate plant began operating in 1930.

It contained the first commercial Dorr-Oliver wet process phosphoric acid units. In this plant phosphate rock is treated with sulphuric acid to liberate phosphoric acid. The phosphoric acid is ammoniated to produce ammonium phosphate fertilizer.

The acid section treats about 900 tons of phosphate rock per day. This is a finely ground concentrate received by rail from Vernal, Utah, purchased from Stauffer Chemical Company. The rock is transferred by pneumatic dust pumps into two 2500 ton storage silos, or to the acid section. About 790 tons per day of 93% sulphuric acid is consumed.

The rock is batch weighed into an 85,000 gal Dorr single tank reactor. Here phosphoric acid and insoluble gypsum are formed.

$$Ca_{10}(PO_4)_6F_2 + 10\,H_2SO_4 + 20H_2O \longrightarrow 10CaSO_4 \cdot 2H_2O + 6H_3PO_4 + 2HF \quad (1)$$

Calcium Fluorapatite (phosphate rock)	Sulphuric Acid	Water	Calcium Sulphate (gypsum)	Phosphoric Acid	Hydrofluoric Acid

Conditions of temperature, concentration of acid, and density of the reaction slurry are carefully controlled to produce a good filtering gypsum and to minimize losses.

The reactor is stirred by six agitators using a total of 380 hp. Slurry overflows to a filter feed tank from which it is pumped to a Bird-Prayon tilting-pan filter, located near the top of the building. The active filtering area is 950 square feet. The filter has a 200 hp vacuum pump and a 150 hp cake drying vacuum pump. The temperature of the reaction slurry is controlled to 75°C by removing heat of reaction in a vacuum cooler. About 12,000 gpm of slurry is circulated through the cooler by a 150 hp elbow pump. Reactor and filter ventilation gases are scrubbed in a cross flow water scrubber to remove fluoride vapors.

Primary production acid, containing 30% P_2O_5 with 1.5% excess sulphuric acid, is settled in a 50,000 gal clarifier.

Clarifier underflow and phosphoric acid washings from the filter are mixed with the sulphuric acid in a fluoride stripper. The mixture is cooled with air spargers to about 100°C, and flows into the reactor. Silicon tetrafluoride evolved from the stripper is scrubbed with water in a two stage plastic absorber vessel to make fluosilicic acid.

$$\underset{\substack{\text{hydrofluoric}\\\text{acid}\\\text{(reaction (1))}}}{6HF} + \underset{\substack{\text{Silica}\\\\\text{(from rock)}}}{SiO_2} \longrightarrow \underset{\substack{\text{fluosilicic}\\\text{acid}}}{H_2SiF_6} + \underset{\text{water}}{2H_2O}$$

$$\underset{\substack{\text{fluosilicic}\\\text{acid}}}{H_2SiF_6} \xrightarrow{\text{heat}} \underset{\substack{\text{silicon tetra-}\\\text{fluoride}}}{SiF_4\ \text{(gas)}} + \underset{\substack{\text{hydrofluoric}\\\text{acid}}}{2HF}$$

$$\underset{\substack{\text{silicon}\\\text{tetrafluorine}}}{3SiF_4} + \underset{\text{water}}{2H_2O} \longrightarrow \underset{\substack{\text{fluosilicic}\\\text{acid}}}{2H_2SiF_6} + \underset{\text{silica}}{SiO_2}$$

Fluosilicic acid is used as the electrolyte in the Lead Refinery and for municipal water fluoridation. Gases from the fluosilicic acid absorber are further scrubbed in a water scrubber followed by a packed spray tower to abate fluoride emissions.

Clarified phosphoric acid is evaporated to 40% P_2O_5 in four horizontal tube lead evaporators and one forced circulation evaporator with a total operating steam use of 30,000 lb. per hour. Evaporated acid is clarified to remove impurities and fed to the 11-55-0 fertilizer granulation units. Clarifier underflow passes to the production acid clarifier.

The fertilizer section contains three Dorr granulation units, which can produce about 220-250 tons per day each.

Each granulation unit is comprised of an ammoniation reactor, a blunger (pugmill), an 8' x 60' drier, a recycle system of belts and elevators, screens, pulverizer, dust cyclones, dust recovery impingers, and abatement scrubbers. About 800,000 cu. ft. per day of natural gas is used for drying.

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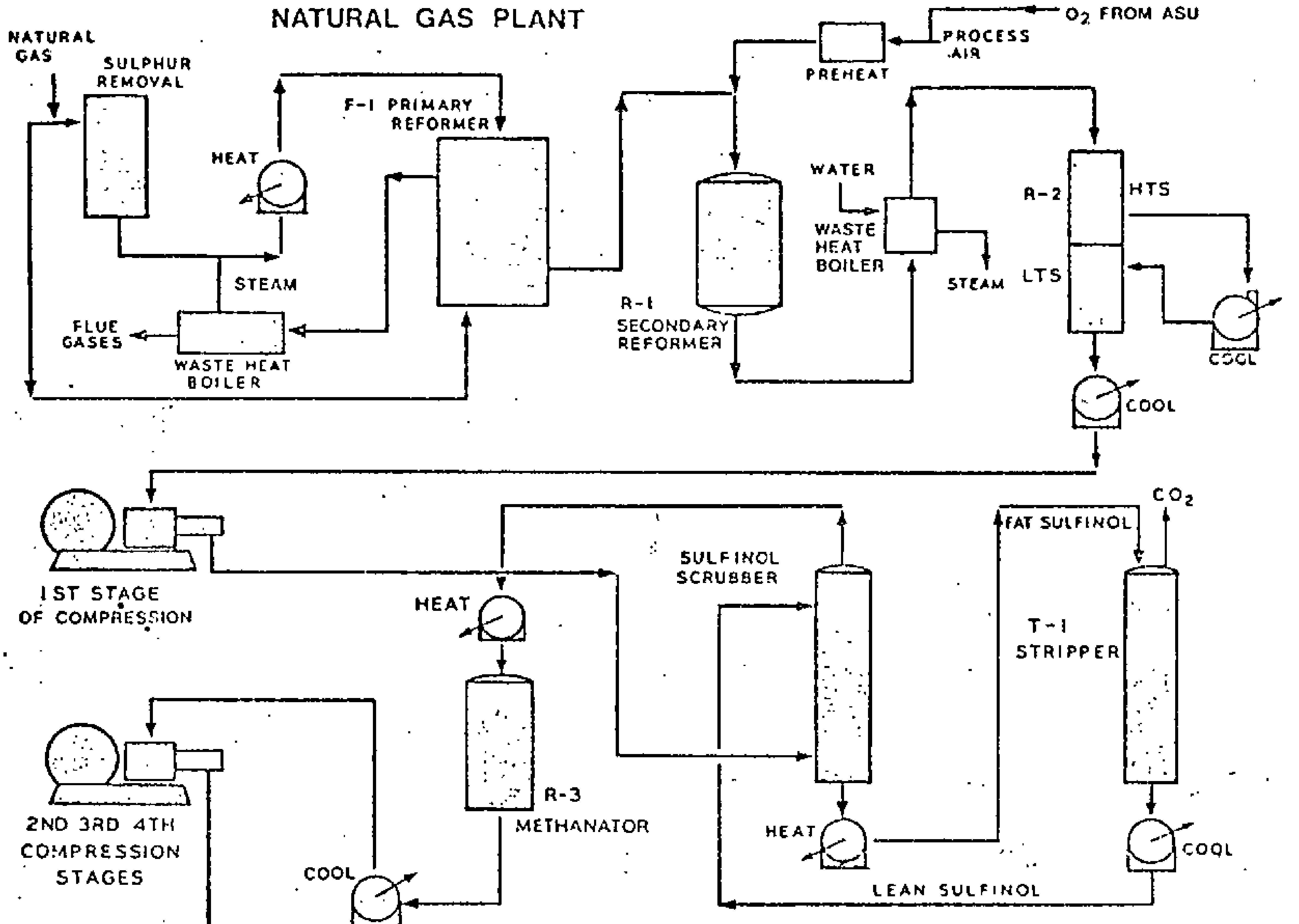
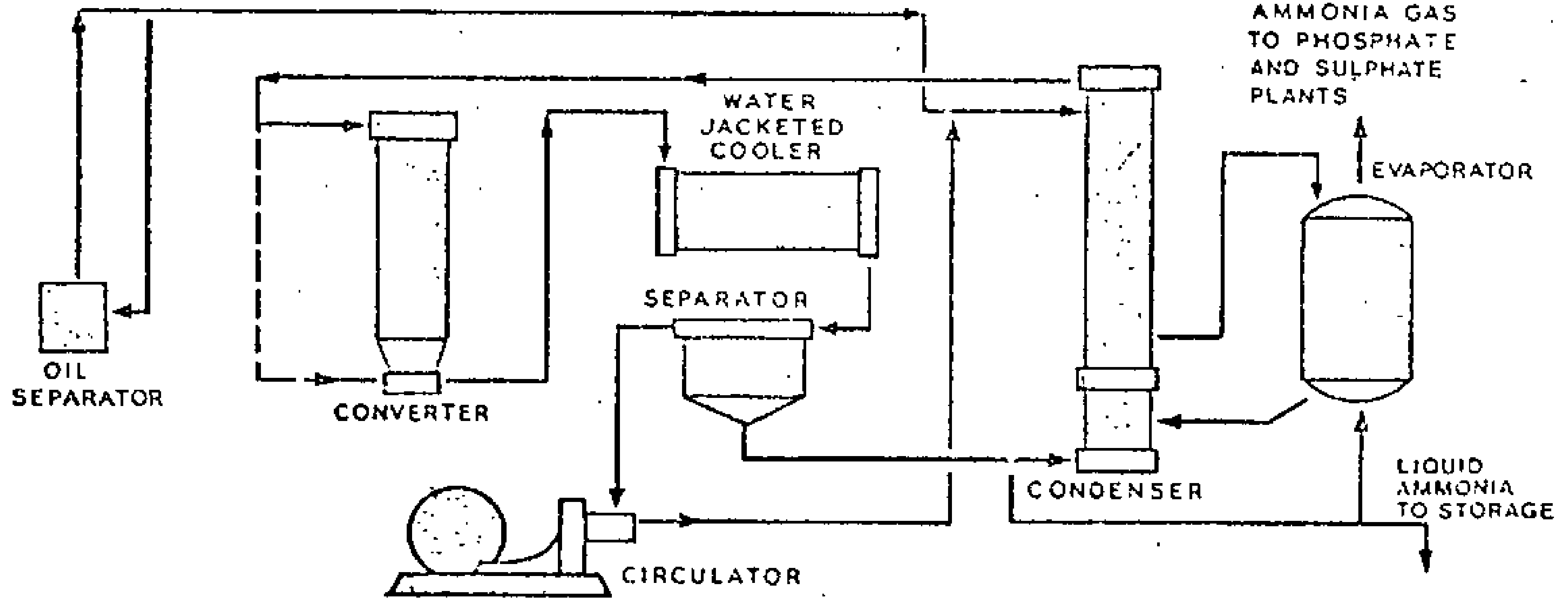
SIMPLIFIED FLOWSHEET
AMMONIA PRODUCTION AND AMMONIUM SULPHATE PLANT

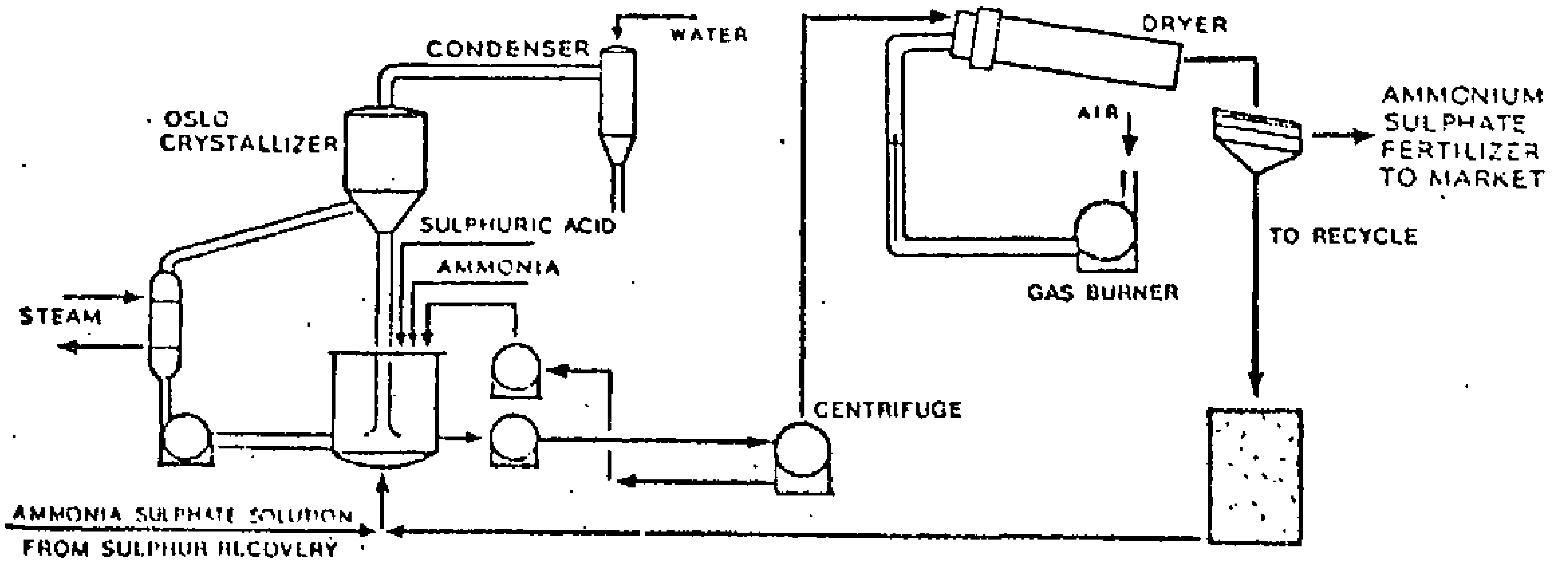
NATURAL GAS PLANT

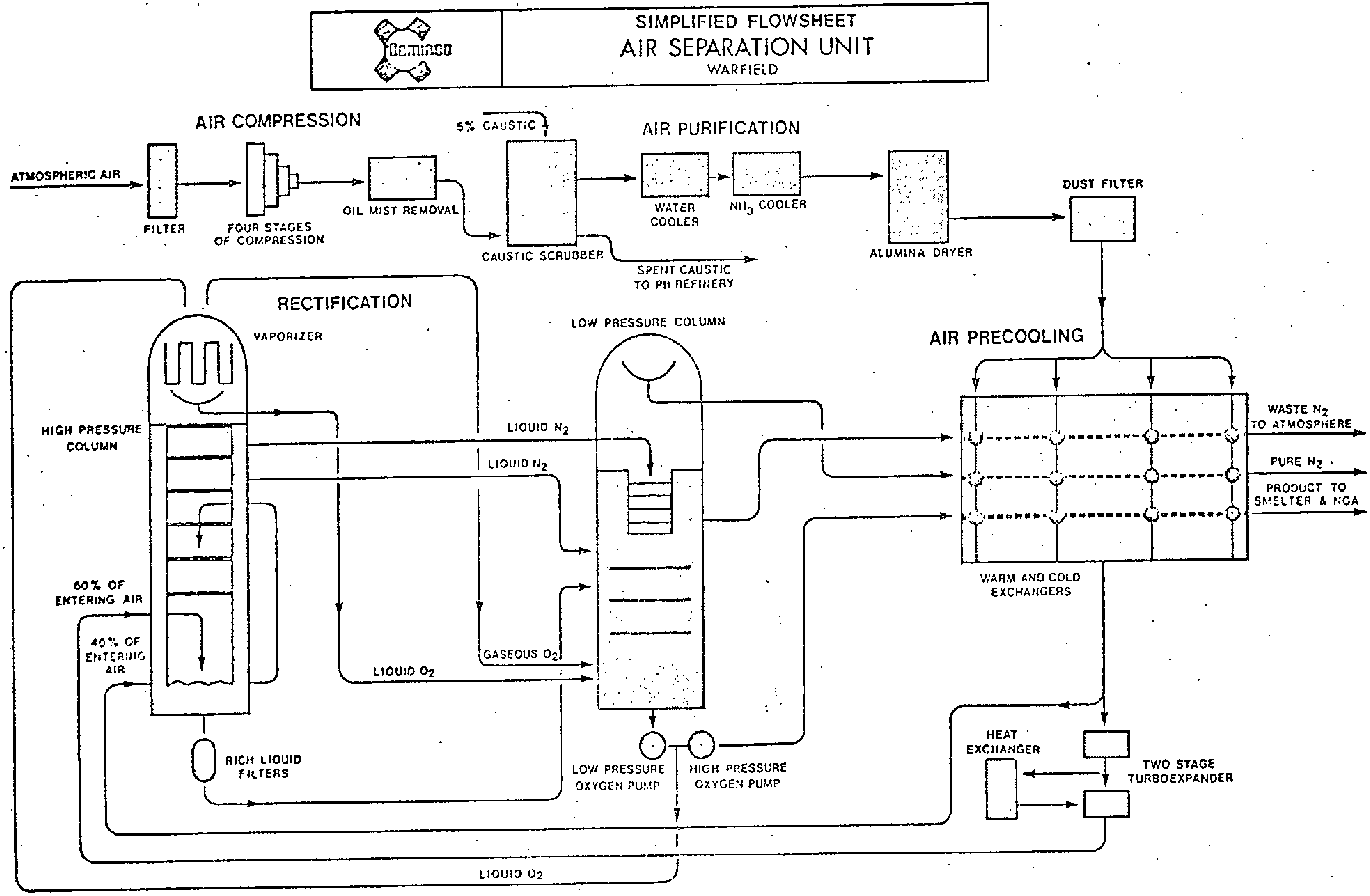


SYNTHESIS AMMONIA PLANT



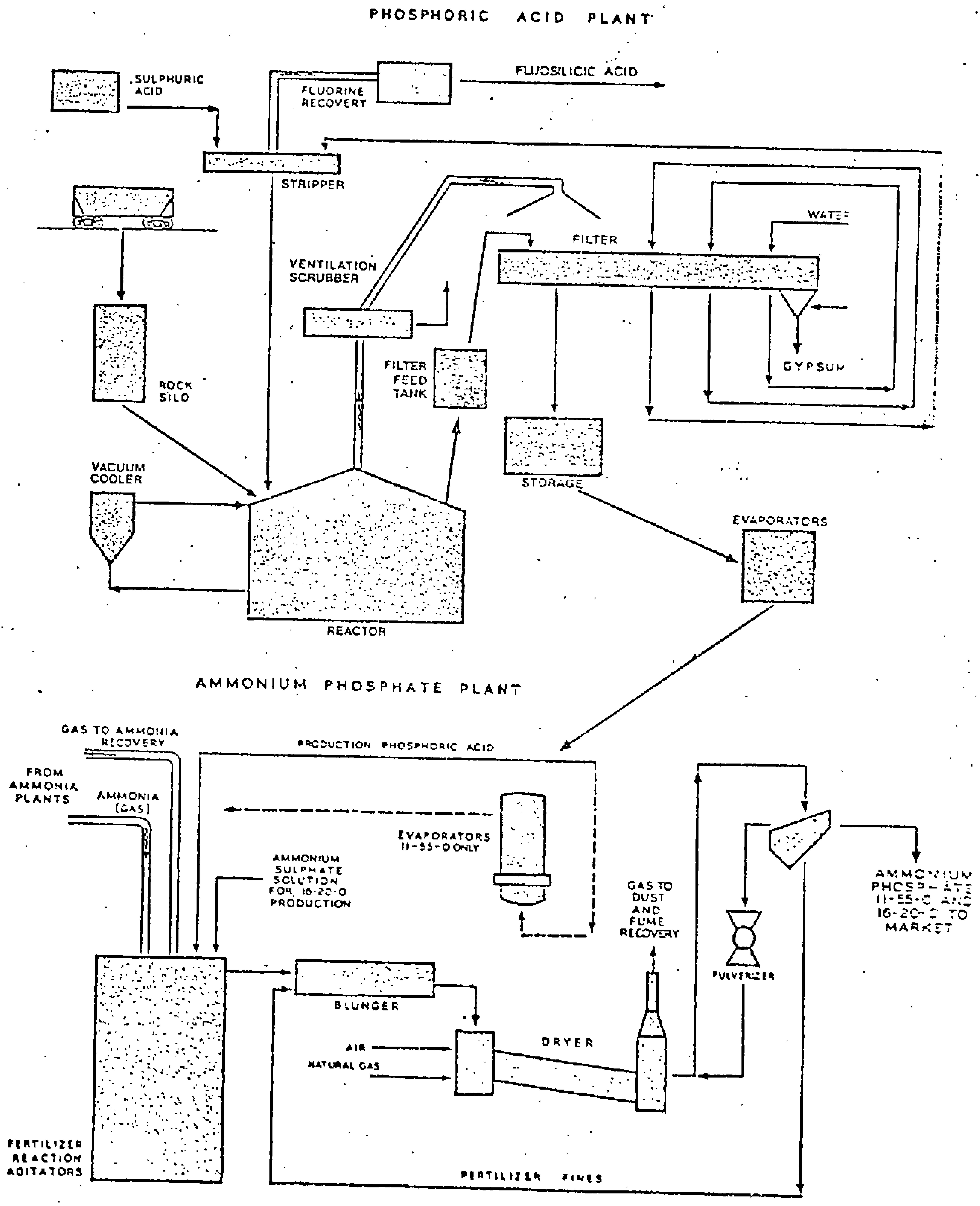
AMMONIUM SULPHATE PLANT




SIMPLIFIED FLOWSHEET
AIR SEPARATION UNIT
WARFIELD

Cominco

SIMPLIFIED FLOWSHEET
PHOSPHATE PLANTS
WARFIELD



Usually two units make 11-55-0, monoammonium phosphate. The third makes 16-20-0, ammonium phosphate-sulphate, which incorporates ammonium sulphate fines from the ammonium sulphate plant.

Ammonium Sulphate Plant

The ammonium sulphate plant was also started in 1930. This plant receives a 40% ammonium sulphate solution pumped from Tadanac. The solution is evaporated to produce a crystalline ammonium sulphate fertilizer, 21-0-0.

The plant contains four crystallizers of 100 tons per day production capacity and one crystallizer of 250 tons capacity. The smaller units operate under vacuum at 70°C, using some second effect steam from the large unit which operates at 105°C. Crystal slurry is circulated through steam heated exchangers to the evaporators. Total operating steam use is 95,000 lbs. per hour. Water and phosphoric acid are added to control nuclei and crystal size. Crystals are separated from mother liquid in horizontal basket centrifuges, which operate on automatic cycles. The crystals are dried of 2 - 3% water in three 6 1/2' x 20' gas fired driers. Dry crystals are screened and about 17% of the product is removed as fines. Some of the fines are redissolved and recrystallized,and some are pneumatically transferred to the 16-20-0 fertilizer unit in the phosphate plant.

Engineering, Maintenance and Construction

Introduction

The Trail Operations of Cominco have been running for over 70 years. Until after the Second World War the location was quite remote, and this required the establishment of a major group of shops for the overhaul and manufacture of equipment. Traditionally, the Company has also done much of its own engineering and construction work, and this has led to the development of two strong engineering organizations in Trail: Trail Operations Engineering and a corporate engineering group known as Cominco Engineering. Today most standard types of equipment are purchased from the major manufacturers, but it has still been found economic to continue to run a large shop complex in support of the maintenance groups in Trail and other operations. More use is being made of specialized consultants for many aspects of engineering but Cominco Engineering still develops nearly all the conceptual engineering and design required for new projects, completes the feasibility and economic studies, and, for many projects, also does the detailed design.

Trail Operations Engineering Organization

There are two main divisions in the Engineering support group in Trail:

1. Maintenance
2. Design and Construction

1. Maintenance

In Maintenance, each of the four activities -- Lead Smelter, Refineries, Zinc, and Chemicals and Fertilizers -- has its own assigned maintenance group under a superintendent. These assigned maintenance groups, which vary from 15 men in the Refineries to 140 men in the Zinc Operations, are responsible for all the normal daily and weekly maintenance in their area. In addition, within Trail Operations organization, there is a central maintenance services group, about 200 men strong, which supplies a number of specialist trades such as bricklayers, and which acts as a backup or reinforcement group to the plant maintenance groups for major plant shutdowns or turnarounds. The maintenance services group also maintains all installations, buildings, and equipment outside plant battery limits.

2. Design and Construction

A design group of about 20 engineers, designers, and draftsmen works directly in support of the Trail Operations. They provide assistance to the maintenance and operating departments, and design most minor betterments and projects. This group has few specialists, and the engineer or designer will take responsibility for a complete job.

Corporate Engineering

Cominco Engineering is a corporate group, located in Trail, which serves all the operations of Cominco. Although organized to function on an arms-length basis, this group is intimately involved in supporting engineering activities at Trail. There are five principal departments or activities in the Cominco Engineering organization: Central Shops, Project Engineering, Design, Construction and Technical Services.

Central Shops

The central shops consist of a foundry, machine shop, steel fabricating shop, motor winding shop and a plastic and rubber shop. The central shops do all the shop overhaul and repair of equipment from the Trail plants. In addition, a very wide range of spare parts are manufactured, either on a routine basis for warehouse stock, or as "one-of" items for a particular piece of equipment. The central shops employ about 240 tradesmen and apprentices. Steel castings of up to 10 tons can be poured. Lathes and boring mills are able to handle almost any component from the plants. The steel fabricating shop manufacturers and repairs bins, hoppers, chutes, tanks, ducts, heat exchangers, structural steel work, and also does boiler repairs. The motor winding shop rewinds and repairs motors of up to 3,500 H.P. and transformers of up to 12,000 KVA, 60 KV. The plastic and rubber shop manufactures and installs a wide range of equipment from items weighing a few ounces up to complete tanks and vessels and tank linings.

Construction

The construction group averages about 150 tradesmen, and occasionally peaks at about 350 men on major projects. Projects vary in size from small revisions to existing plants, up to complete new plants in the $1,000,000 to $20,000,000 range.

Project Engineering

The project engineering group does most of the preliminary and conceptual engineeing work, including the development of feasibility reports and estimates for the appropriation of funds for significant new projects. Projects are structured on a team or task force basis, with a project engineer supported by an operating representative, a design representative, and a construction representative for every active project. The team, under the direction of the project engineer or superintendent is responsible for the design, construction and project management of the entire project.

Design Engineering

The corporate design engineering office of about 80 engineers, designers, and draftsmen is divided into specialist groups: mechanical, electrical, civil, piping and pressure vessels, heating and ventilating, and process control (instrumentation). This office does all the design work for major projects, and also provides help and back-up to the Trail design office in specialist fields.

Technical Services

The technical services group provides expertise in the areas of materials engineering and non-destructive testing, analysis of equipment failures, material for any type of service or environment, and a consulting service in all aspects of materials handling problems.

Management Systems

Both Trail Operations Engineering and Cominco Engineering have a full range of systems for financial and technical control and management of their activities. In maintenance, cost records and technical histories are kept on all equipment. A preventive maintenance system covers all key equipment. Backlog is monitored continuously, and the crew sizes adjusted to conform with the long-term trends in the work load. In construction, costs are kept updated daily, and the exact state of actual expenditure versus the detailed estimate is kept under constant review. The central shops operate a job costing system, and the exact cost breakdown of every job is given to the plants. In the project and design groups, all jobs are listed in the backlog file, which is updated weekly to show the status of the job, its current priority, and the engineering and design time charged to it both for the week, and for the whole job to date.

Provision and Training of Tradesmen and Engineers

A very thorough apprenticeship training scheme for tradesmen has been in operation for over 40 years. The majority of the tradesmen in Trail served their apprenticeship with Cominco. Currently there are over 100 apprentices in the Trail Operations, in 21 different trades. Selection and training is rigorous and practically all apprentices graduate with a Provincial Certificate with the Inter-provincial Seal.

Engineers are recruited from universities across Canada, and are given an eighteen-month "Engineer-in-Training" program. During this time they spend about three months in each of the major departments, followed by two to five years in a junior engineer's appointment in a particular department. Progression is then normally into a senior engineer's appointment in a technical field, or into a middle management position in one of the engineering or operating groups.

Relatorio Mensal de Andamento ao CETEM

DIVISAO: DIMETE DATA: 30 de março de 1982

Projeto: Estagio STI/CIDA - Tecnologia de Fornos de Leito Fluidizado
Canadian Centre for Mineral and Energy Technology - CANMET
555 Booth Street, Ottawa, ONTARIO, K1A 0G1, CANADA

Relatorio no. 6

De: Jorge L.L. Brandao,
CANMET, Room 270.

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Science and Technology
Science et Technologie

Your No Votre référence

Our file Notre référence

Relatorio no. 6: Março/82

No decorrer do mes de março foram realizadas as sequintes atividados:

1 - Termino da montagem do equipamento definitivo.

2 - Testes preliminares com o equipamento.

3 - Preparaçao dos testes de ustulaçao com o concentrado de zinco de Morro Agudo: aguardando análise granulométrica.

4 - Visita técnica:

New Brunswick Research and Productivity Council, RPC
Fredericton, New Brunswick: 25-26 Março, 1982
Ver "Trip Report" anexo.

5 - Termino do estagio: Ver "Summary Report" anexo com fotos do equipamento.

NOTA: Devido a problemas com o equipamento de cromatografia gasosa nao foi possivel prosseguir com programa de testes.

Ottawa, 30 de março de 1982

Jorge L.L. Brandao

Jorge L.L. Brandao
eng. metalurgico.

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TRIP REPORT

New Brunswick Research and Productivity Council (RPC)
Fredericton, New Brunswick - March 25-26, 1982

The purpose of this trip was to meet with Dr. Igor Wilkomersky who has been involved with RPC's Sulphation Roast-Leach (SRL) Process. Dr. Wilkomersky is responsible for the design of RPC's 14 in. reactor where sulphation roast tests are carried out.

During my first visit to RPC, last January, Dr. Wildomersky was away in Chile where he had been working as professor at the University of Concepcion. Also, during that time there were no tests being carried out in that reactor. Presently RPC's 14-in. reactor is being used for sulphation roast tests of the Caribou Zinc Ore (Anaconda) which contains over 40% zinc and enough pyrite to sustain and even generate heat during the runs, which is controlled by water spraying at the top of the reactor.

After a good deal of exchange of information between Dr. Wilkomersky and myself concerning fluid bed reactors, Dr. Roy Boorman, head of Mineral Development and Processing Department, showed me a report he is sending to CIDA on Transfer of Technology to Brazil, involving RPC's Sulphation Roast-Leach Process. Dr. Boorman is likely to meet with myself again at CETEM (Centre for Mineral Technology) in Rio, in the coming April or May, subject still to CIDA's approval. His main interest in Brazil is to promote RPC's SRL process with Brazilian mining companies which he hopes to accomplish via CETEM.

Ottawa, March 30, 1982

Jorge L.L. Brandao

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Metallurgical Engineer

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SUMMARY REPORT

by Jorge L.L. Brandao

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CII.D.A. Sponsored Programme on Fluid Bed Reactor Technology at CANMET, EMR.

This programme started in early October, 1981 and ends on March 30, 1982.

During the programme I had the opportunity to visit various research centres, university laboratories and industrial operations and met with leading authorities in the field of fluid bed technology. In early October I attended a 3-day course in New Orleans, U.S.A., given by Dr. Frederick Zenz for which I received a certificate. This course was fundamental to my programme at CANMET since it gave me a better perspective of what there is available in terms of information and literature as well as an up-to-date view of research in fluid bed technology around the world.

The main purpose of the programme was to become knowledgeable in the building of laboratory and pilot fluid bed reactors for roasting sulphide ores and concentrates.

In order to have a "feeling" of the mechanisms involved I started out by making a small "Pyrex" reactor for studies at room temperature. Modifications were made and improvements introduced after almost every visit to other research facilities and industrial operations. After about 4 months the studies on the small "Pyrex" model were completed (see Figure 1) and, in the meantime, parts for a larger continuous feed reactor, for studies at reaction temperatures (up to about 1100°C), were being designed (some as early as December, 1981), and, after ordering and receiving all necessary materials, those were sent to CANMET's Machine Shop and glassblower to be constructed. The waiting period was extremely length y and it was only in early March that the fluid bed reactor unit, comprising a three-zone furnace, a pre-heating furnace, a settler, primary and secondary cyclones, a filter and a pH-monitoring scrubber with recirculating caustic solution was finally completely assembled (see Figures 2,3,4 & 5). During the first three weeks of March, tests were carried out for "trouble-shooting" pruposes and a few modifications were introduced.

.../2.

Finally, on March 22nd the equipment was ready for a final check and at least one actual test with a Brazilian Zinc Concentrate, received from CETEM last February. However, due to problems with the gas chromotograph equipment for analysing the exiting gases, the test which had been started in the afternoon on March 22 had to be cancelled.

Unfortunately, there was no time left to run any more tests and it will be up to CANMET/MSL to determine whether or not the equipment will be used in the future. In case the decision is favourable I will always be available at CETEM to answer any questions and give any explanations concerning its proper use so that this equipment may be of use to others.

Ottawa, March 30, 1982

Jorge L.L. Brandao.

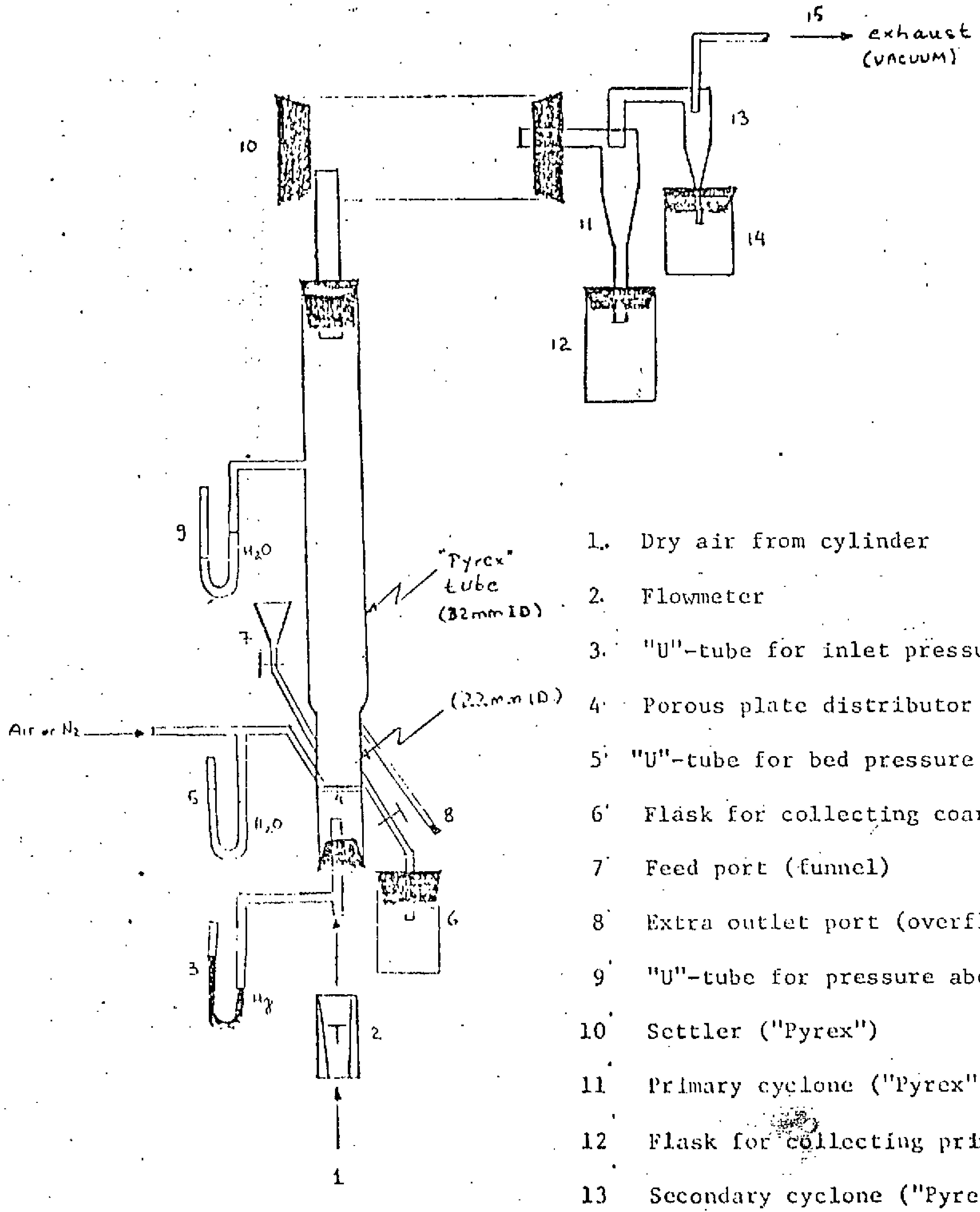
Jorge L. L. Brandao,
Metallurgical Engineer,
CANMET/MSL.

c.c. Marlene Saydeh (CIDA)
Marie Paige (CIDA c/o M. Saydeh)
Peter Pint
John Dutrizac
Mike Campbell

JLLB/jo

Figure 1: Sketch of "Pyrex" Reactor for Studies at Room Temperature

Sketch of the equipment:



1. Dry air from cylinder
2. Flowmeter
3. "U"-tube for inlet pressure (mercury)
4. Porous plate distributor (silica glass)
5. "U"-tube for bed pressure (water)
6. Flask for collecting coarse particles
7. Feed port (funnel)
8. Extra outlet port (overflow); closed
9. "U"-tube for pressure above bed (water)
10. Settler ("Pyrex")
11. Primary cyclone ("Pyrex")
12. Flask for collecting primary fines
13. Secondary cyclone ("Pyrex")
14. Flask for collecting secondary fines
15. To vacuum line

NOTE: Heating can be accomplished by wrapping a heating tape around the reactor tube (maximum temperature obtained: 200°C)

Figure 2: General View of Fluid Bed Reactor Set-up

From left to right:

- pH-monitored caustic scrubber unit
- pre-heating box furnace and temperature monitoring read-outs; vaccum pump (behind furnace)
- feeding system (vibrating trough)
- gas cleaning system (settler, primary & secondary cyclones)
- fluid bed reactor in three zone "Lindberg" furnace
- bottom discharge
- "Lindberg" furnace temperature controls

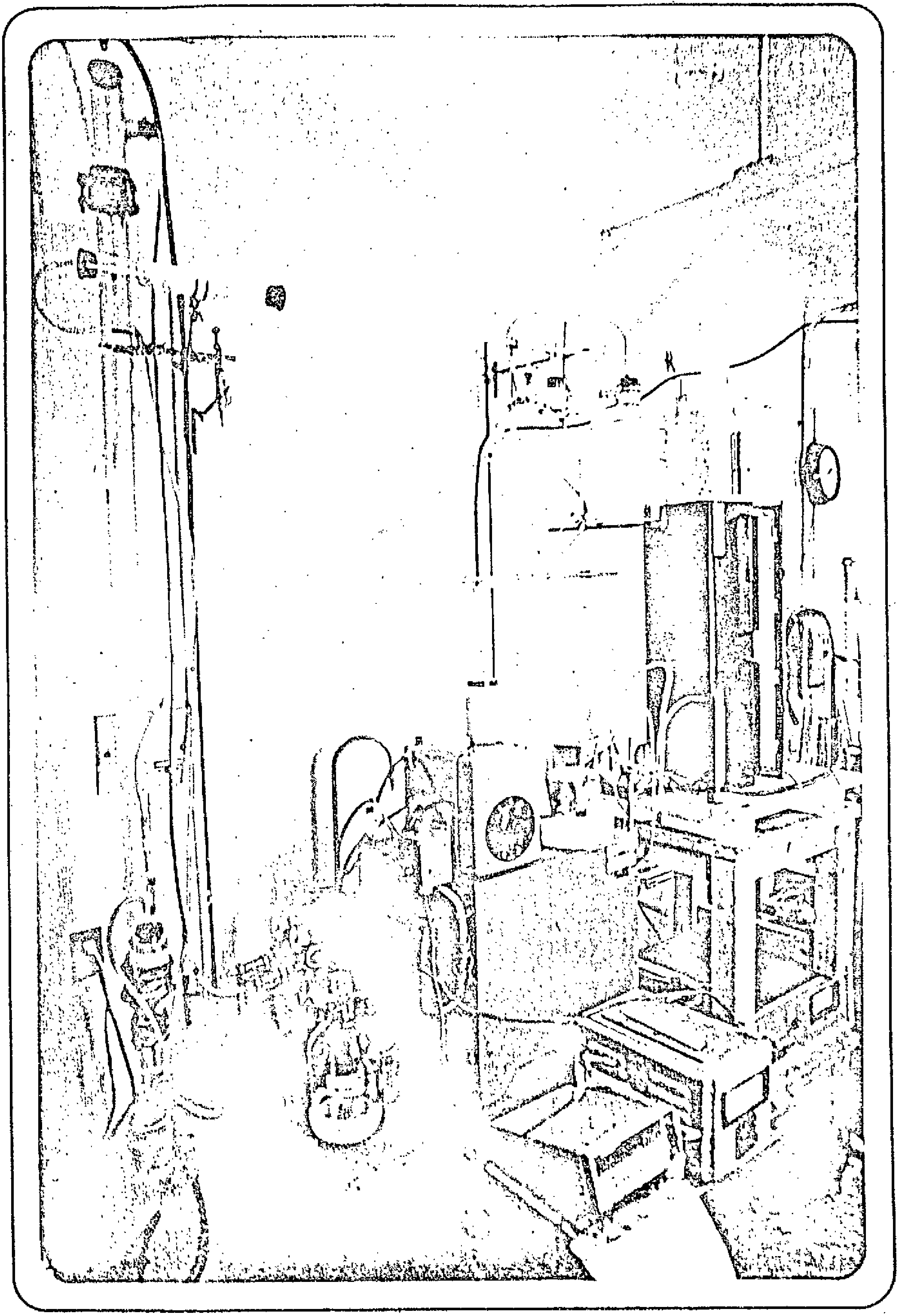


Figure 3: Front view of the equipment

from left to right:

- scrubber and superfines filter
- pre-heater "Variac"
- feeder
- settler and cyclones
- reacting air rotameter
- three-zone furnace (open) with silica glass reactor

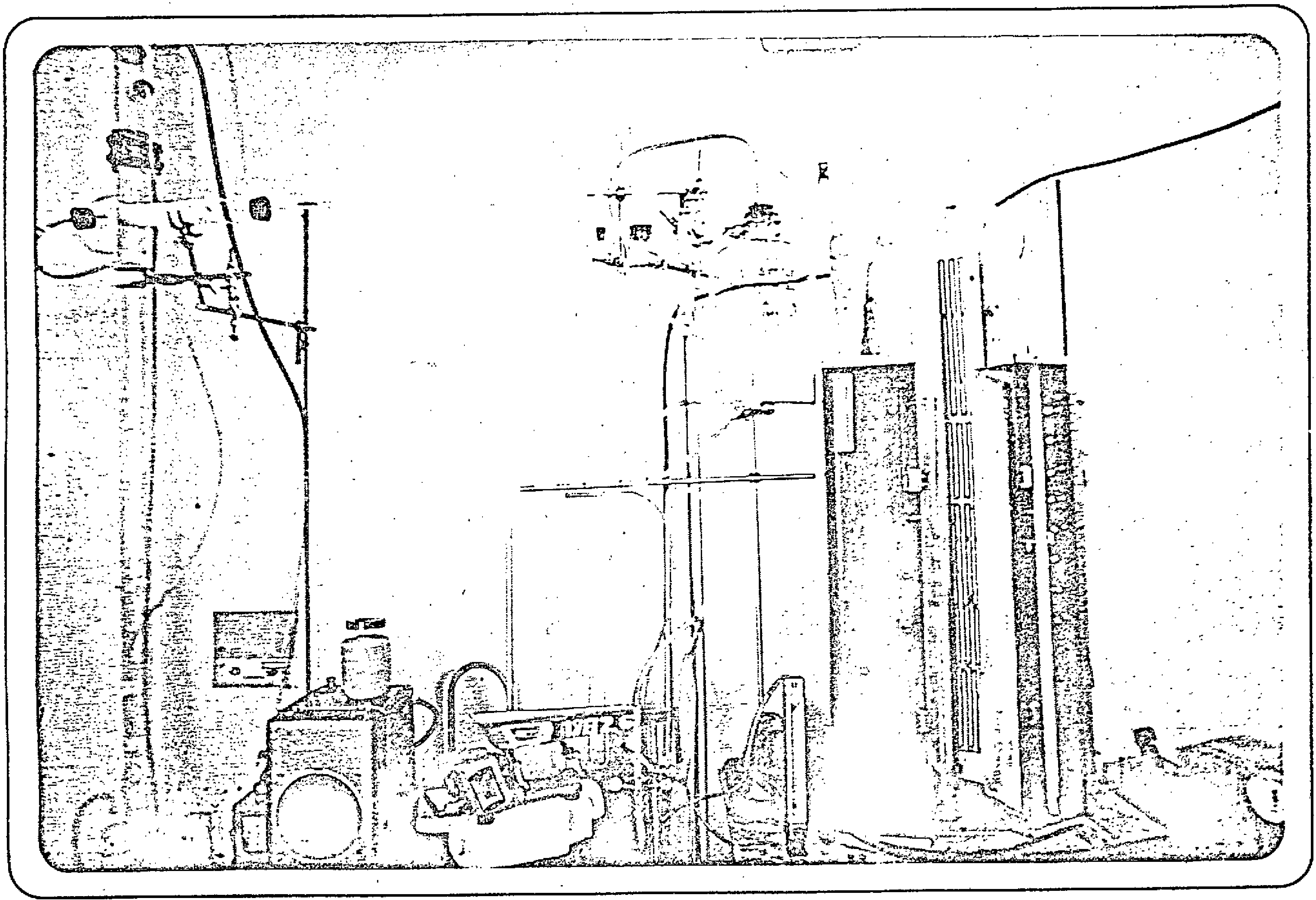


Figure 4: Gas Cleaning System

from left to right:

- secondary cyclone
- primary cyclone
- settler (depicting thermocouple arrangement)

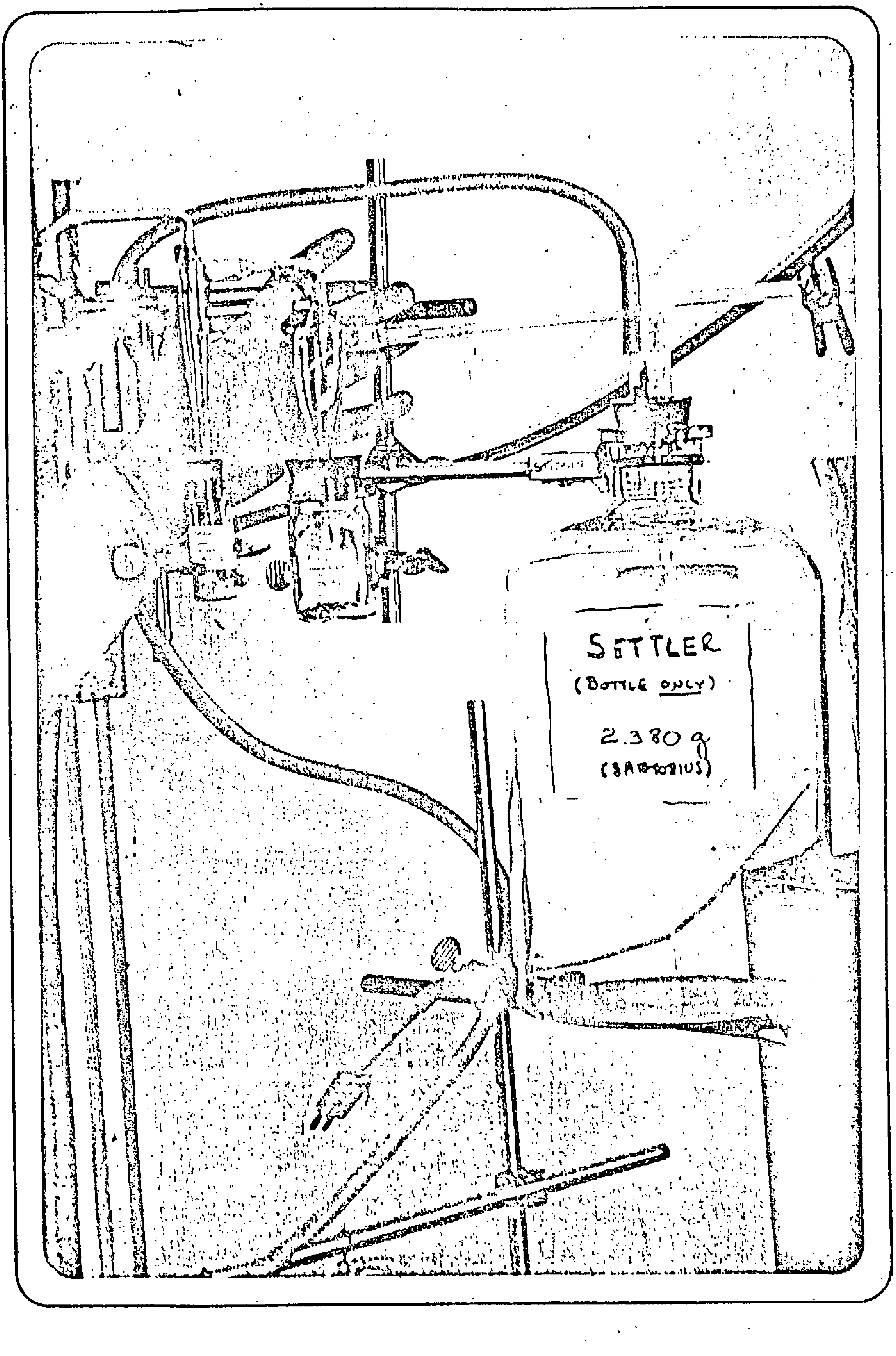
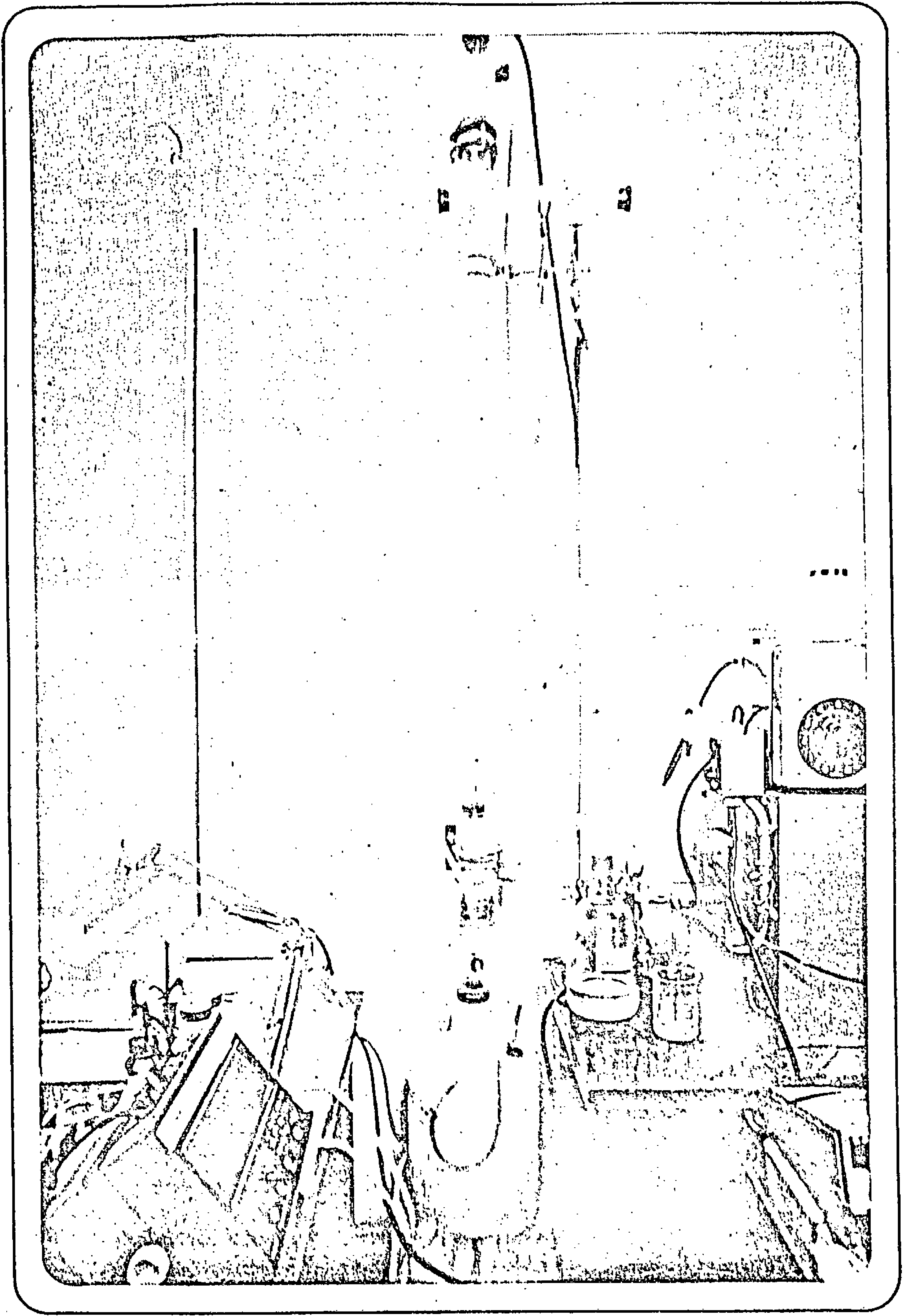

SETTLER
(BOTTLE ONLY)
2.380 g
(SARTORIUS)

Figure 5: pH-Monitored Caustic Scrubber and Superfines Filter

from left to right:

- pH-meter and peristaltic pump
- caustic solution container
- scrubber and filter (at the top)
- part of pre-heating furnace and scrubbing solution bottle
- part of vacuum pump (behind bottle of solution)



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Este relatório é uma compilação dos relatórios enviados mensalmente à Divisão de Metalurgia Extrativa do CETEM durante o período do estágio.

Um relatório final está sendo elaborado, a ser enviado à STI/MIC, CNPq e CPRM (uma cópia deste relatório ficará à disposição na biblioteca do CETEM.

Rio de Janeiro, 04 de março de 1982

JORGE L. L. BRANDÃO
Engº Metalúrgico