

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

RESEARCH ON SMELT-WATER REACTIONS

1. Second Activity Report of Project Co-ordinator, IPC Project 2419
2. Progress Report of The Babcock & Wilcox Company
3. Progress Report of Combustion Engineering, Inc.

June 24, 1964

THE INSTITUTE OF PAPER CHEMISTRY  
Appleton, Wisconsin

RESEARCH ON SMELT-WATER REACTIONS

SECOND ACTIVITY REPORT OF PROJECT CO-ORDINATOR

Project 2419

June 24, 1964

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THE INSTITUTE OF PAPER CHEMISTRY  
Appleton, Wisconsin

RESEARCH ON SMELT-WATER REACTIONS

SECOND ACTIVITY REPORT OF PROJECT CO-ORDINATOR

This report covers activities of the Smelt-Water Research Project from the March 4, 1964, date of the initial Activity Report through May 12, at which time the Babcock & Wilcox and the Combustion Engineering research groups were asked to undertake the preparation of their initial progress reports. These reports from the B & W and C-E research laboratories are submitted herewith.

A good start is being made on a complex and difficult problem. Small-scale experiments by Combustion Engineering have produced laboratory explosions by the controlled addition of a few milliliters of water to molten smelt, and have confirmed indications from previous work of the effect of sulfidity in increasing explosive violence. Study and sampling of conditions in an operating furnace by B & W are expected to yield a better understanding of the complex possibilities for reaction in the lower portion of the recovery furnace. Thermodynamic calculations by B & W of possible furnace reactions will benefit from recently published computer techniques for mathematical exploration of equilibrium conditions among the furnace reactants.

Additional background information has been developed to relate to the project. The bibliographic staff of The Institute of Paper Chemistry has compiled a group of approximately 160 references bearing on various aspects of the smelt-water explosion problem. Sets of this material have been furnished to B & W and C-E, and it is planned to assemble all of the pertinent literature in an annotated bibliography to be distributed to the sponsor companies.

On March 17, H. S. Gardner attended meetings of the Black Liquor Recovery Boiler Advisory Committee and its Subcommittee on Emergency Shut-Down Procedures, held in Atlanta, Georgia. Information gained at these meetings and through contact with these committees is helpful in adding to the general knowledge being accumulated regarding the explosion problem.

On March 18, a visit was made to the Bruceton, Pennsylvania, Field Station of the U. S. Bureau of Mines. The visiting group consisted of Messrs. Nelson, Plumley, and Norton of Combustion Engineering, together with H. S. Gardner. Earlier plans for attendance by H. P. Markant of B & W were cancelled because of last-minute developments. A tour of the Bruceton laboratories and discussions with the scientific staff brought out a number of ideas for equipment and measurement techniques in the study of explosions.

Visits were made on April 2 and 3, respectively, to the research laboratories of B & W and C-E for discussions between H. S. Gardner and the technical staffs on more specific plans for the research programs.

On May 4 and 5, visits were made to the Wilmington, Delaware, area for discussions with explosives experts of Hercules Powder Company and the Du Pont company. The visiting group included Messrs. Anderson, Ekman, and Matty of B & W, Messrs. Nelson and Plumley of C-E, and H. S. Gardner. The experts at Hercules, as well as those at Du Pont, felt confident that the damage patterns and types of explosions experienced in recovery furnaces were the result of rapid gas evolution or the deflagration of a mixture of explosive gases. They felt that a true detonation would produce such a severe shock that the furnace might well be demolished. The Hercules representative has subsequently sent us additional information on flame and explosion detection and suppression.

The Du Pont group expressed two important concepts which they have learned through bitter experience. First, they try as far as possible to take discretionary judgment in hazardous operations out of the hands of the operators. Second, when carrying out a hazardous operation, equipment and procedures are so designed that the quantity of hazardous material is held at a minimum.

Inspections of the Du Pont Hazardous Materials Laboratory and their Explosives Research Laboratory brought us in contact with an impressive array of equipment and measurement facilities.

At 2:30 a.m. on May 24, a recovery boiler suddenly exploded at Potlatch Forests, Lewiston, Idaho. One man was very slightly injured, and the boiler damage was relatively light in comparison with prior major explosions. Earlier established understandings regarding future explosions were followed, and the field organization of Combustion Engineering promptly notified the C-E research group of the explosion. With excellent co-operation from the Potlatch management, Wharton Nelson and H. S. Gardner inspected the boiler from May 27 through May 30. Much useful information was gained. In the light of our present need for a much clearer understanding of the mechanism of recovery furnace explosions, we cannot offer at this time a satisfactory explanation for the Potlatch incident. Although we do not have definitive answers, we feel that the first-hand experience at the site of the incident was well worth while. We feel that if another explosion should unfortunately occur, it should be similarly observed as promptly as possible by a research team.

In summary, we are impressed with the seriousness of the explosion hazard in recovery furnace operation. The last two explosions at Thurso (November 30) and Potlatch (May 24) each occurred at a time when furnace operation was considered to be normal and potentially hazardous conditions were not recognized. In each case, there appears to have been the possibility of introducing water or dilute solution into the furnace bed. We have ample laboratory evidence from both B & W and C-E that smelt and water, under certain conditions, can produce explosions. Our problem is to achieve an understanding of the way, or ways, in which explosive conditions can develop within a furnace bed. The present programs appear to be our best hope of eventually learning enough about these explosions to be able to prevent them.



Howard S. Gardner  
Project Co-ordinator  
The Institute of Paper Chemistry  
Appleton, Wisconsin

PROGRESS REPORT

FOR THE PERIOD OF DECEMBER 1, 1963 TO MAY 12, 1964

TO

H. S. GARINER, PROJECT COORDINATOR,  
THE INSTITUTE OF PAPER CHEMISTRY

INVESTIGATION OF SMELT-WATER REACTIONS

Submitted by:

THE BABCOCK & WILCOX COMPANY  
RESEARCH CENTER  
ALLIANCE, OHIO

June 1, 1964

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PROGRESS REPORT OF THE BABCOCK & WILCOX COMPANY  
ACTIVITIES ON INVESTIGATION OF SMELT-WATER REACTIONS

INTRODUCTION

The problem of occasional severe explosions in Kraft recovery boilers has been a source of concern to the pulp and paper industry for many years. As a result a joint research effort is being undertaken by The Babcock & Wilcox Company and Combustion Engineering, Inc. under the sponsorship of the companies forming the Smelt-Water Research Group. At the present time, it is generally agreed that the explosions take place as a result of the interaction of the smelt bed with water; what interactions take place are not as yet clearly understood. Thus there is some evidence of a chemical reaction between water and smelt that in some manner results in an explosion; at the same time there is evidence that a purely physical encapsulation of water in smelt can lead to the subsequent release of steam with explosive violence. Whatever the mechanism, it is important to determine the relative contribution of each to the over-all problem.

By agreement, The Babcock & Wilcox Company studies are concerned primarily with the chemical aspects of the problem. It is the objective of this work to determine the chemical reactions which take place between the smelt-combustion gas system with water and/or steam from the point of view of determining what explosive reactions may take place. It is a further objective to determine the critical parameters affecting the explosive reactions in order to outline the preventive measures to be taken.

Active work on this phase began April 6, 1964 and this report is a summary of the accomplishments through May 12.



## APPROACH

Our approach is based on the philosophy that we must understand what processes are taking place in order to control or eliminate the explosion problem. In the initial phases of this work we are concerned primarily with (1) obtaining background information from our files concerning circumstances surrounding individual explosion incidents as well as reviewing previous experimental work in the area, (2) obtaining preliminary field data on gas composition, smelt composition and temperatures in operating units, (3) carrying out thermodynamic equilibrium studies in order to determine what chemical species might be expected, and (4) designing experiments and experimental apparatus which will allow kinetic studies to be made on smelt-water reactions.

## STATUS

### Background Studies

To date the background information concerning the B&W experimental work<sup>(1)</sup> has been reviewed and the results of all of the experimental tests have been obtained from our files, including the 30 odd tests not included in the literature. As yet, these results have not been critically reviewed, although preliminarily it appears that most of the explosions obtained can be explained on the basis of the hydrogen-oxygen explosive reaction. One test using sodium tetraborate generated steam only, in contrast to sharp explosions obtained with sodium sulfide. We have started reviewing our files on actual field explosions, but have no comments at this time.

### Field Studies

Arrangements have been made to obtain liquor, char, gas, and smelt samples at the D. M. Bare Company the week of May 18th. These are to serve as guide lines

for future sampling and will be analyzed for the common constituents only, although a qualitative mass spectrometer analysis for unusual constituents will be made on the gas samples if possible.

### Chemical Equilibrium Studies

The basic chemical thermodynamic data ( $\Delta F$ ,  $\Delta H$ ,  $\Delta S$ ) for all of the species of interest have been obtained and critically examined. All data appear to be consistent except for a slight discrepancy in the  $H_{298}$  for  $\text{Na}_2\text{SO}_4(\text{V})$  and a major discrepancy in  $S_{298}$  for  $\text{Na}_2\text{S}$ . At present it appears that May's<sup>(2)</sup> value of 18.6 calories/ $(^\circ\text{K})(\text{mole})$  is low and we shall tentatively use the value 23.2 calories/ $(^\circ\text{K})(\text{mole})$ , obtained by Latimer's estimation method,<sup>(3)</sup> in our calculations until a better experimental value is available.

The available methods for computer calculation of complex equilibria have been reviewed and the free energy minimization method, proposed by White, Johnson and Dantzig<sup>(4)</sup> and further described by Oliver, Stephanou and Baier<sup>(5)</sup> appears most applicable and will be used.

### Experimental Studies

Most of the readily available literature has been reviewed by abstract and 15 reprints of original papers which are pertinent to the reactions of sodium sulfide, sodium sulfate, and sodium carbonate have been ordered.

Preliminary efforts have been made in designing laboratory apparatus which will allow kinetic studies of the smelt-water reaction. Initial studies, aimed at identifying the reaction products, can be carried out in several ways, one of which involves a percolation of water vapor through molten smelt. Zelezny<sup>(6)</sup> has studied the reaction of water vapor with molten aluminum in this manner and his apparatus is shown in Figure 1. Although he claimed to have obtained kinetic

data, it is difficult to determine the basis on which he tested his data and this leaves an element of uncertainty. As an initial, preliminary design, we propose an apparatus that would allow a fresh liquid surface to be in contact with the steam at all times, thus giving a basis for calculating the kinetics on the basis of a known surface area. We emphasize that this design, shown in Figure 2, is subject to modification.

#### WORK PLANNED FOR NEXT PERIOD AND EXPECTED PROGRESS

It is anticipated that the survey of our files for pertinent information covering explosion incidents will be virtually completed by August 31. All other literature survey work will also have been completed. Furthermore, gas, smelt, and char samples will have been obtained and analyzed. It is expected that the computer program for the thermodynamic calculations will have been written, de-bugged, and checked out on known systems, and should be near completion for the system under consideration. The laboratory apparatus will have been designed and a start will have been made in its construction.

#### EXPENDITURES (APPROXIMATE TO MAY 12, 1964)

Labor	\$2,267
Overhead	3,628
Materials*	307.72
	<hr/>
Total Expenditures	\$6,202.72

\*Includes travel expenses

A cost control chart showing actual and anticipated expenditures is attached.

CHA:vlr

Submitted by: C. H. Anderson  
C. H. Anderson  
Research Chemist

Approved by: W. A. Keilbaugh  
W. A. Keilbaugh, Chief  
Chemical Section

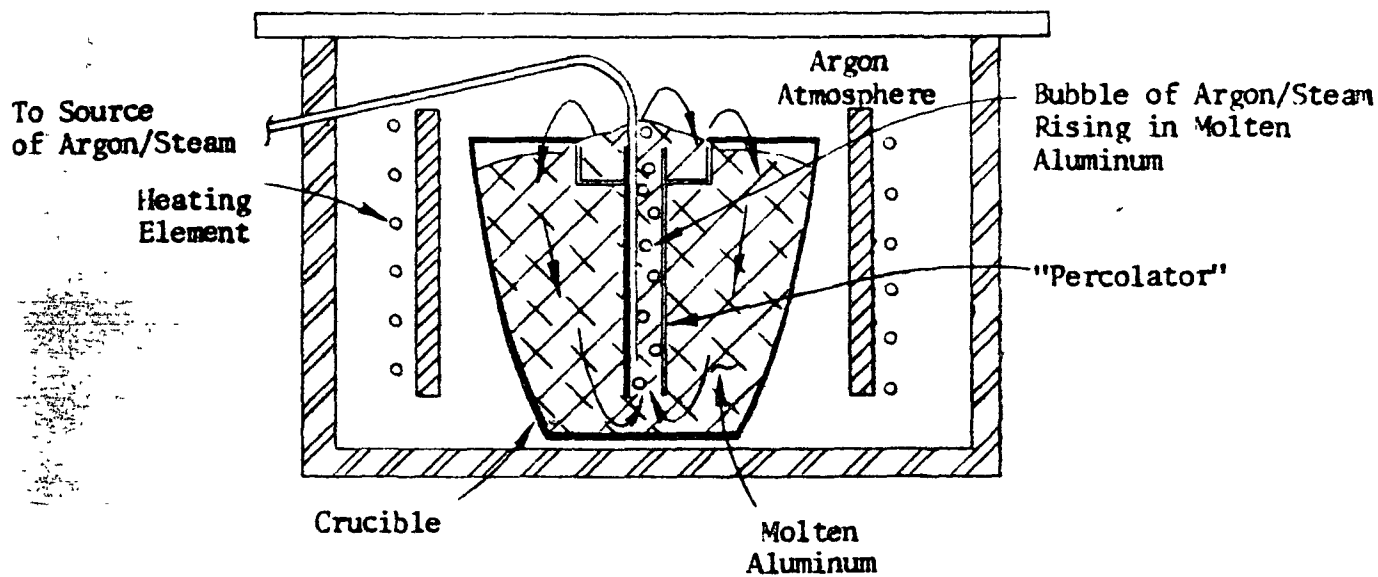
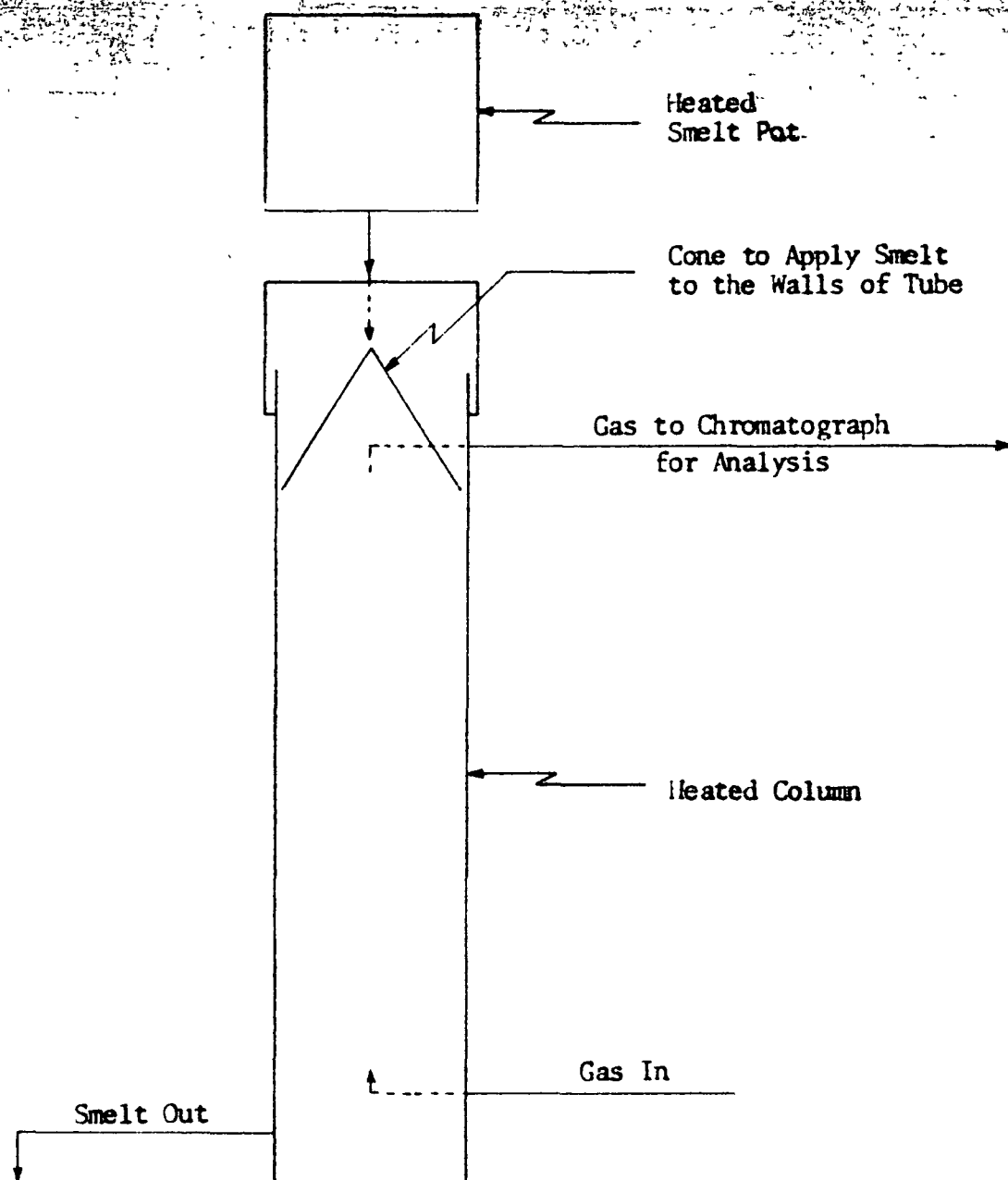
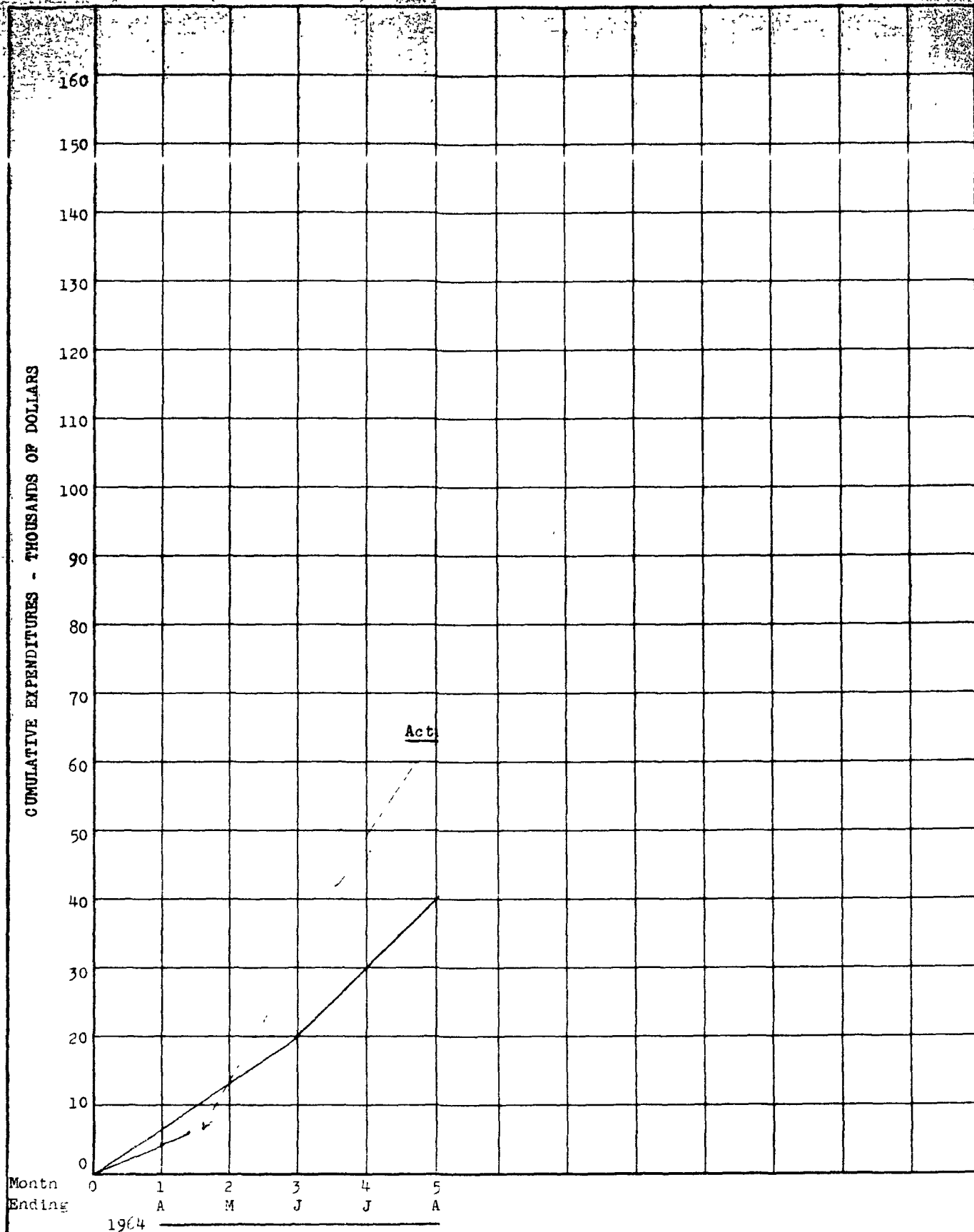


FIG. 1. SCHEMATIC DIAGRAM OF APPARATUS FOR CIRCULATION OF MOLTEN ALUMINUM (REF. 6)



Note: All parts in contact with molten smelt to be either silicon carbide or graphite.

FIG. 2. WETTED WALL COLUMN FOR STUDYING SMLT - WATER (STEAM) REACTION RATES



SUBJECT ANTICIPATED EXPENDITURE VS TIME CUR  
INVESTIGATION OF SMELT WATER REACTI  
FOR FOURDRINIER KRAFT INSTITUTE

(BIMONTHLY) PROGRESS REPORT  
COVERING WORK TO MAY 15, 1964

TO

DR. H. S. GARDNER, PROJECT COORDINATOR  
THE INSTITUTE OF PAPER CHEMISTRY

Submitted by

COMBUSTION ENGINEERING, INC.  
WINDSOR, CONNECTICUT

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In all the work so far considered very little real quantitative data was obtained. The products of reaction, the temperatures and pressures involved and general destructive power are all qualitative observations inadequately supported by quantitative data. Combustion Engineering, Inc. has felt that a detailed scientific study of such explosions under controlled conditions is essential to obtain sufficient knowledge of the problem to allow the development of a practical solution.

### Scope

The above considerations led to a natural division between the chemical and physical aspects and the emphasis of the C-E investigation is on the physical factors involved. In the early planning for this project it was thought that such studies might be carried out in an actual unit. An old unit was located which was near retirement but it would have had to be reassembled in a new location. The cost of relocation plus rebuilding after each explosion made this operation impractical. A pilot furnace was considered and again expense of building and operating such a unit made the operation unfavorable. In both an actual unit and a pilot plant the control of operating conditions and monitoring of events within the bed during explosions is quite difficult.

It was finally decided that a laboratory reactor was the appropriate device for studying explosions---at least in the initial stage. The C-E reactor (a 4 ft. closed spherical barricade) is undergoing final design after consultation with the Bureau of Mines, du Pont, and Hercules. Appropriate instrumentation, including a high speed multi-channel recorder and high speed photography is planned. Closed-circuit television is available for remote visual operation. The design of this reactor was decided upon to allow more accurate evaluation of pressure developed, the effects of controlled atmosphere and a more positive identification and analysis of reaction products. In addition considerable scale-up of explosions (and suggested remedial measures) would be possible in a vessel of this size.

There is no intent to repeat the work of previous investigators in this field. The effects of smelt composition, for example, have already been well covered. Repetition will be only to the extent that it serves to calibrate properly the apparatus and methods used in this study. Upon completion of the reactor priority in experimental work should be given to determining whether smelt-water reactions are combustible in nature. A number of quenches will therefore be made in air and inert atmosphere. Nitrogen rather than steam will be used for



initial inerting experiments due to the chance of possible steam-smelt reactions at some temperature levels. Quenches with water introduced beneath molten smelt (to simulate a bed leak) will be compared in violence and product composition with those in which water is applied above the smelt, which would approximate a water-wall tube leak above the bed.

Active work on the phase of the work covered in this report began about February 1, 1964 and results through May 15 are included. Subsequent reports will be issued every two months.

### Procedure

The best method to learn how to eliminate smelt-water explosions seemed to be to study the variables which affect them on reaction of small batches of smelt with water under controlled conditions. During construction of the barricade a series of preliminary experiments involving addition of water to small crucibles of molten smelt and smelt to water by various means is under way. The quenching experiments have been carried out in a barricade described later.

In general, these initial experiments have involved variation of the relative amounts of smelt and water and geometry of the quench vessel. Small quantities of smelt have been used in order to minimize the costs and danger to personnel. Pure chemicals were melted together in an inert atmosphere to make typical smelts of known composition.

Included in this discussion are the data on experimental quenches involving the bringing together of a relatively large amount of molten smelt or other molten material with a relatively small amount of water. There was at least a 100 to 1 smelt to water ratio. The molten charge averaged about 200 grams. It was found generally that the ability of a smelt to explode and the rapidity with which the smelt exploded after contact with water, increased with sulfidity increase. There was a measurable time delay between the addition of water and the explosion in most runs. The explosions appeared to occur most readily when the smelt temperature approached the freezing point, e.g. as the viscosity increased. Efforts are now under way to determine the relationship of viscosity, hardness and compression strength of smelt to its explosive tendency in contact with water.

### Experimental

The synthetic smelt was made by heating dried flake sodium sulfide with anhydrous sodium carbonate under nitrogen in a

4 inch carbon crucible contained in a steel ladle to permit easy pouring. The first 22 runs were made by pouring the molten smelt at 1550°F. or other material into one of the cups containing water (Figures 1-3). Subsequent runs have been made by injecting water through a stainless steel tube into the heating crucible under the smelt. (Fig. 4). A newly designed apparatus (Fig. 5) will also lead the water under the smelt.

These quenches have been carried out within a 4 ft. by 8 ft. barricade of 5/8" plywood. This structure (Fig. 6) is equipped with two safety glass windows on opposite sides, a blow out door and a hood for concentrating any gases evolved during quenching experiments. The vessel containing smelt is ordinarily located directly under this hood during quenches. Gas sampling has been done by means of a probe under the hood near the mouth of the reaction vessel. An evacuated sample bottle is attached to the probe to collect the gas. Analyses were made on a Perkin-Elmer 154-D Gas Chromatograph using silica gel and molecular sieve columns.

## Results & Discussion

Since the emphasis of the C-E investigation is, by contract, on the physical aspects of the smelt-water reaction, it is logical that any known physical explanation of explosion should be checked by experiment for this specific interaction between smelt and water. The ability of some molten materials to encapsulate a quantity of water for sufficient time so as to result in an eruption of steam with some violence is documented by experience in aluminum and steel industries. Fire prevention manuals customarily warn against attempts at extinguishing fires with water near molten salt baths.

Initial experiments, then, were designed to evaluate the encapsulation of water by smelt as an explosion mechanism.

The temperature of molten material at the start of these quenches was between 1550 and 1600°F. and it can be seen from the data that those smelts containing the most sulfide proved to be most subject to explosion. It was not possible to observe an explosion with either aluminum or pure carbonate under similar conditions. The minimum quantity of water needed for explosions does not appear to be limited, (Runs 6, 17, 18, 23, 33), but complete coverage or encapsulation of some quantity of water by the smelt is necessary for the combination to result in explosion.

The loudest and most violent explosion occurred when the greater quantity of water was encapsulated with smelt of the same composition (Runs 14-17). However, even a small quantity of water resulted in a vigorous explosion when the sulfide

concentration of the smelt was increased (Runs 24, 26, 28).

Injection of the water under the smelt seems to be the most dependable method of obtaining explosions. The use of a 2" crucible for the smelt (Run 32) resulted in a relatively vigorous reaction in which the carbon crucible was shattered. The smaller diameter container of course allowed an increase in depth of smelt which could account for the apparent violence of the reaction.

Calculation based on the shattering of the crucible in run 32 indicate that an internal pressure of approximately 600 psi was necessary to cause this damage. A recorded thermocouple measurement during run 33 shows a stepwise temperature drop at each addition of water. However, the initial addition caused a temperature increase. While still unconfirmed this is an indication of an initial exothermic reaction.

### Summary

Experimental work to date tends to confirm observations from earlier C.E. and B & W work, that the tendency for explosion to occur seems to increase with increasing sulfide content. The changes in hydrogen content in the later runs suggest that hydrogen may be consumed in the reaction but the total is too low to constitute proof that the explosion is due to combustion or ignition of hydrogen. The percents of hydrogen found in all cases have been very low. This checks with the very small proportion of water (0.3 ml) required for explosion in some cases.

### Future Work

Future work will include hardness and compressibility tests on castings of smelts of varying composition. These castings have been made in a 4 inch carbon crucible of 3/4 inch diameter. Viscosity measuring techniques are being developed for the same smelt compositions. Comparison of these physical properties with explosibility of various smelts will be made.

(continued on Page 6)

Specific areas of future investigation are as follows:

- (1) Addition of water
  - (a) Quantity
  - (b) Rate
  - (c) Temperature
  - (d) Direction (above or below smelt)
- (2) Addition of solutions
  - (a) Green Liquor
  - (b) Black Liquor
  - (c) Surface active agents
- (3) Smelt composition
  - (a) Minor activating ingredients
    - (i) NaOH 0.1 - 5%
    - (ii) NaCl 0.5 - 10%
  - (b) Major changes
    - (i) Similar viscosities
    - (ii) Necessity of sulfide for explosion variation

The effect of variation of atmosphere will be made as accurately as possible on this barricade. Effective controlled atmosphere tests must await construction of the spherical barricade.

Submitted by

*A. L. Plumley (by m)*  
DR. A. L. PLUMLEY  
Research Chemist

ALP:ccp  
6-16-64

EXPERIMENTAL QUENCHES

<u>RUN</u>	<u>CHARGE</u>	<u>WATER</u>	<u>CONTAINER</u>	<u>RESULTS</u>	<u>% H<sub>2</sub></u>	<u>% CO<sub>2</sub></u>	<u>% CO</u>
1	50g Anhyd Na <sub>2</sub> CO <sub>3</sub>	~5 ml	A	Water Evap.	-	-	
2	150g Anhyd Na <sub>2</sub> CO <sub>3</sub>	~5 ml	A	Water Evap.	-	-	
3	150g Smelt 13% Na <sub>2</sub> S	~5 ml	A	Water bubbled Through Molten Smelt	-	-	
4	150g Smelt ~25% Na <sub>2</sub> S	~5 ml	A	Small Pops Spattering of Smelt	-	-	
5	250g Anhyd Na <sub>2</sub> CO <sub>3</sub>	~5 ml	A	Water bubbled Through Molten Na <sub>2</sub> CO <sub>3</sub>	-	-	
6	100g Smelt ~21% Na <sub>2</sub> S	0.3 ml	B <sub>1</sub>	Explosion Rifle Crack Some Smelt Blown 20 ft. away	-	-	
7	150g Smelt ~22% Na <sub>2</sub> S	0.5 ml	B <sub>2</sub>	Loud Pops Spattering Some Smelt Scattered 3-4 ft.	-	-	
8	150g Smelt ~20% Na <sub>2</sub> S	0.8 ml	B <sub>3</sub>	Some Spattering Water Bubbled Through Molten Smelt	-	-	
9	150g Aluminum	0.3 ml	B <sub>1</sub>	No Explosion	-	-	
10	150g Aluminum (Hotter)	0.3 ml	B <sub>1</sub>	No Explosion Hissing and Channeling around edges of Aluminum	-	-	

# EXPERIMENTAL QUENCHES

<u>RUN</u>	<u>CHARGE</u>	<u>WATER</u>	<u>CONTAINER</u>	<u>RESULTS</u>	<u>% H<sub>2</sub></u>	<u>% CO<sub>2</sub></u>	<u>% CO</u>
11	150g Na <sub>2</sub> CO <sub>3</sub>	0.3 ml	Small undercut hole B <sub>1</sub>	Water bubbled out through molten carbonate	-	-	
12	150g Na <sub>2</sub> CO <sub>3</sub>	0.4 ml	Sealed glass capsule wired in B	No Explosion Some Channeling	-	-	
13	150g Na <sub>2</sub> CO <sub>3</sub>	1.0 ml	Sealed glass capsule in B	No Explosion Elaborate Channeling	-	-	
14	150g Smelt 20% Na <sub>2</sub> S	1.0 ml	Sealed glass capsule in B	Loud Explosion Container Emptied	-	-	
15	150g Smelt 18% Na <sub>2</sub> S	1.0 ml	Sealed glass capsule in B	Loud Explosion Container Emptied	-	-	
16	150g Smelt 21% Na <sub>2</sub> S	0.3 ml	Sealed glass capsule in B	No Explosion, capsule not completely covered, smelt mostly yellow; some red in water path	-	-	
17	150g Smelt approx. 20% Na <sub>2</sub> S	0.3 ml	Sealed glass capsule in B	No Explosion, channeling red color surrounding path of water--- remainder of smelt yellow	-	-	
18	150g Smelt approx. 20% Na <sub>2</sub> S	2.0 ml	Sand layer - in B $\frac{1}{4}$ inch	Series of pops Smelt very dark red, porous and expanded.	-	-	

## EXPERIMENTAL QUENCHES

<u>RUN</u>	<u>CHARGE</u>	<u>WATER</u>	<u>CONTAINER</u>	<u>RESULTS</u>	<u>% H<sub>2</sub></u>	<u>% CO<sub>2</sub></u>	<u>% CO</u>
25*	300g Anhydrous Na <sub>2</sub> CO <sub>3</sub>	~50 ml added in 5 ml increments	Heating Crucible	No Explosion Water boiled. Some spattering	-	-	-
26*	350g Smelt 8.8% Na <sub>2</sub> S	~30 ml added in 5 ml incre- ments ~10 ml in 1 ml increments	Heating Crucible	Explosion - more moderate than run 24 ~20-30 sec. delay	Before Exp. .10 Gas sampling probe broke	-	-
27*	350g Aluminum	~80 ml added in 5 ml incre- ments	Heating Crucible	No Explosion Aluminum bubbles	.04	-	-
28*	350g Smelt ~27% Na <sub>2</sub> S	~2 ml but injection not completed	Heating Crucible	Violent Explosion most sensitive so far---injection tube inverted and twisted around ladle handle <1 sec. delay	Before Exp. .004 After None	3.7 - -	- - -
29*	350g Aluminum in iron ladle	~80 ml in 5 ml increments	Heating Ladle	No Explosion Large Aluminum bubbles Some spattering of Al	-	-	-
30	400g Aluminum poured into Cup B	~100 ml in 5 ml increments	Injected into B	No Explosion Some spattering Large Aluminum Bubbles	Before Exp. None After .02	- - -	- - -

<u>RUN</u>	<u>CHARGE</u>	<u>WATER</u>	<u>CONTAINER</u>	<u>RESULTS</u>	<u>% H<sub>2</sub></u>	<u>% CO<sub>2</sub></u>	<u>% CO</u>
31*	350g Smelt ~27.1% Na <sub>2</sub> S	~15 ml slurry of lg/600 ml Fluorocarbon <i>Sulfuric Acid Agent</i>	Heating Crucible	Loud Explosion Some Time Delay 15-20 sec.	Before Exp. .005 After Exp. None	16	-
32*	100g Smelt ~28% Na <sub>2</sub> S	~3 ml Single 2 ml increment - explosion at start of second addition	Small 2" Heating Crucible	Violent Explosion Carbon Crucible Shattered - explosion occurred as flame on edges went out	Before Water None Before Exp. .10 After Exp. .02	2.8 4.2 C.43	-
33*	200g Smelt ~28% Na <sub>2</sub> S	20 ml in 2 ml increments	Heating Crucible	Series of 7 or 8 moderate explosions at each addition of water til crucible was emptied. Some green liquor splattering.	Before Exp. .20 After 1st Experiment .46	2.5 1.5	.04 .02

\*Water injected under molten material in heating crucible.



CUP A

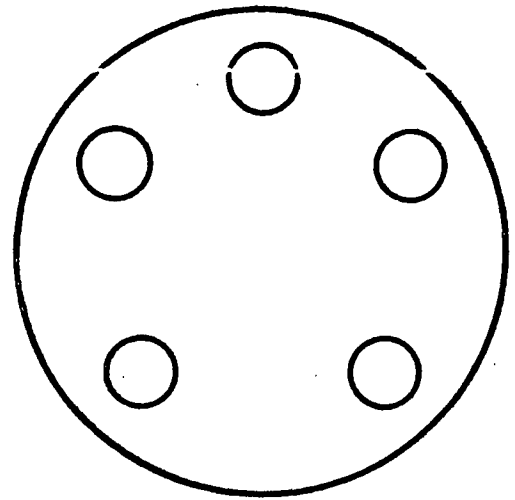
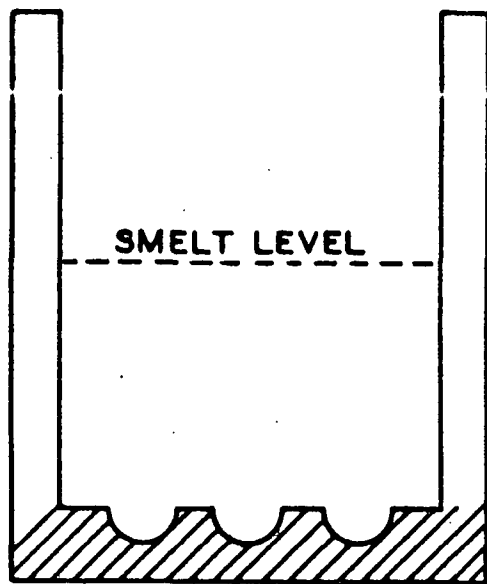


FIG. 1

CUP B

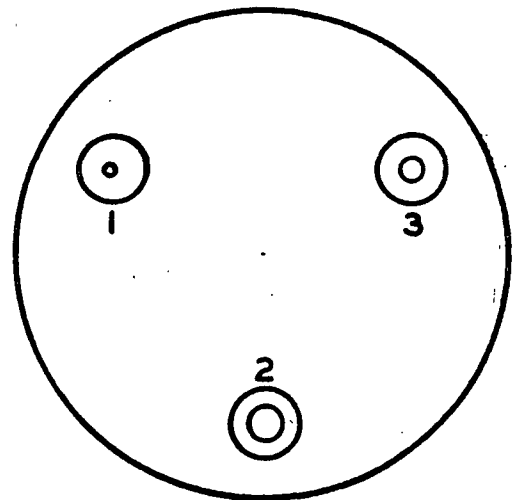
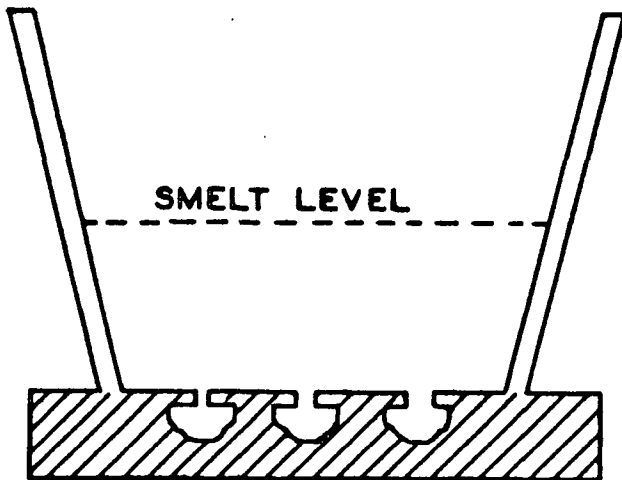


FIG. 2

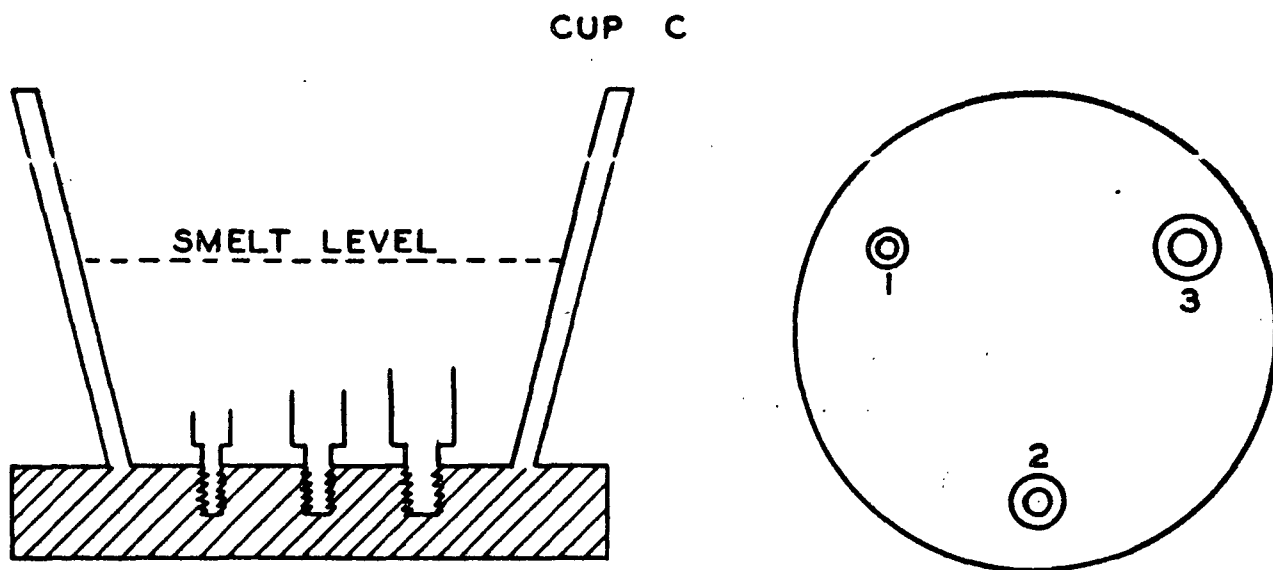


FIG. 3

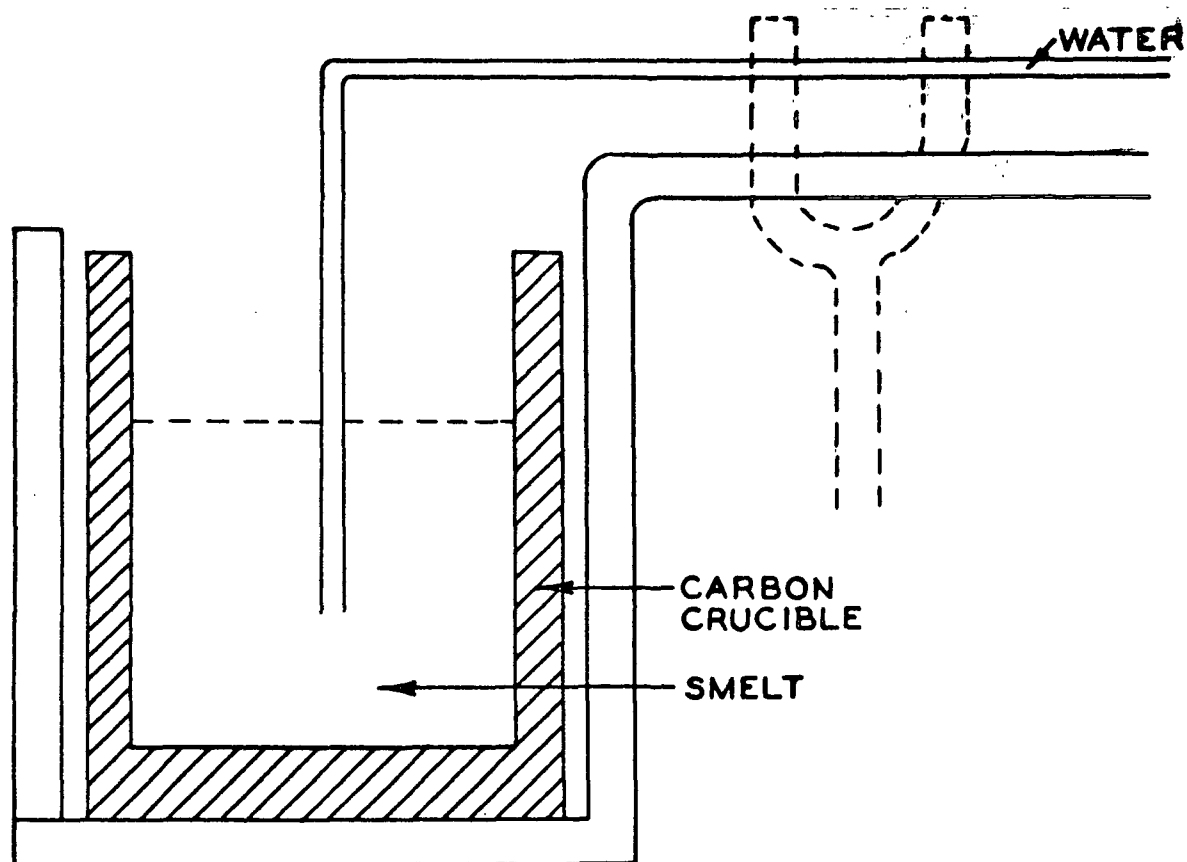


FIG. 4

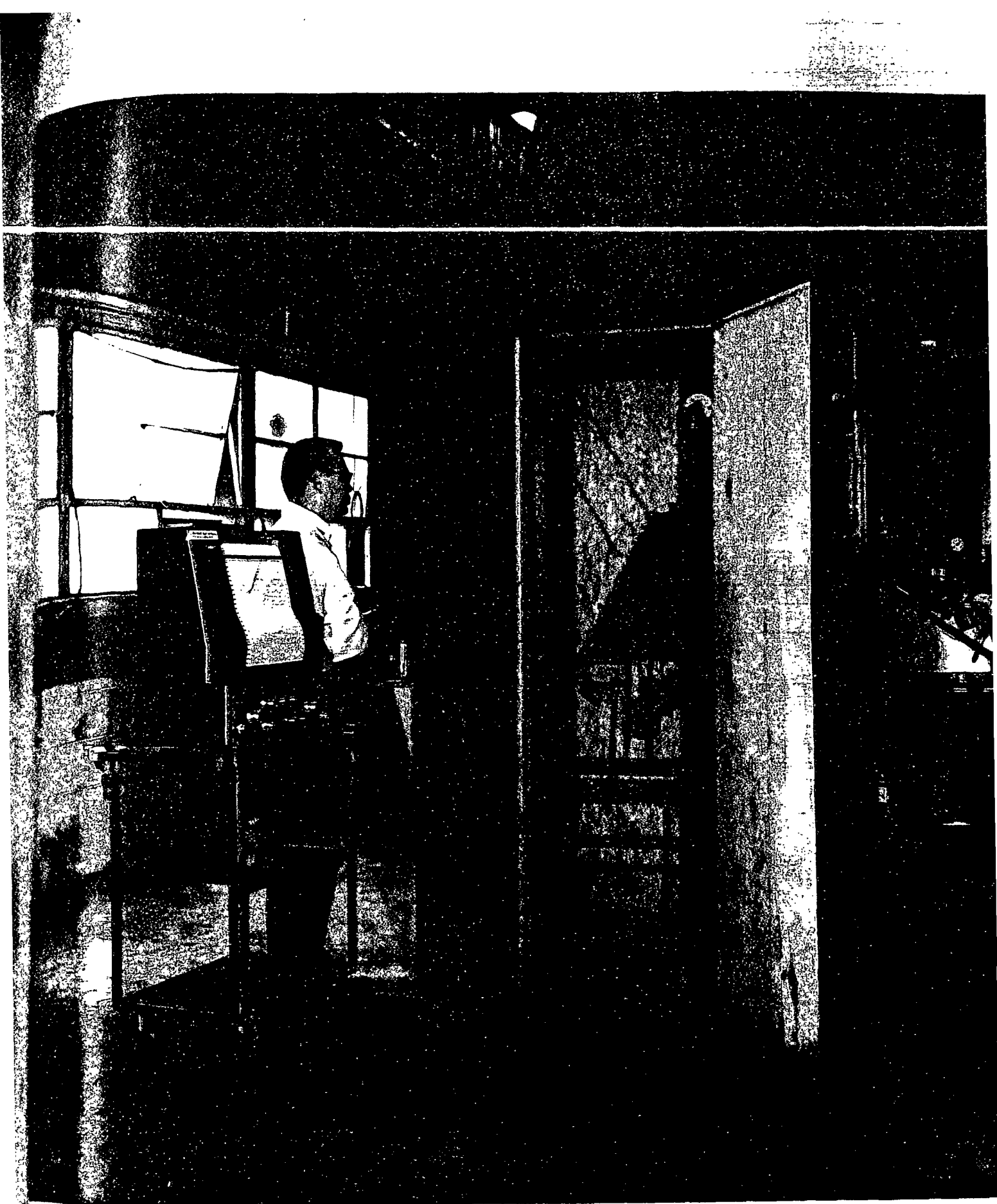


FIGURE 6