

ENCLOSURE (B)2

STUDIES ON MATERIALS
OF ANTI-CORROSIVE REACTION TUBE
FOR PRODUCING HYDROGEN PEROXIDE
BY HYDROLYSIS OF AMMONIUM PERSULPHATE

by
CHEM. ENG. LIEUT.
J. UETA

Research Period: 1944-1945

Prepared for and Reviewed with Author
by U. S. Naval Technical Mission to Japan

December 1945

ENCLOSURE (B)2

LIST OF TABLES
AND ILLUSTRATIONS

Figure 1(B)2	Reaction Tube	Page 33
Figure 2(B)2	Pure Lead (0.016% Sb)	Page 34
Figure 3(B)2	Lead Containing 2% Antimony	Page 34
Figure 4(B)2	Lead Containing 0.2% Antimony	Page 34
Figure 5(B)2	Phase Diagram	Page 34
Plate I(B)2	Flow Sheet of 40 T/M 30 Wt% H ₂ O ₂ Apparatus	

ENCLOSURE (B)2

SUMMARY

The object of this study was to determine suitable materials and working methods for the reaction tube with a long life.

Significant results were:

Lead which contains from 0.2% to 2.0% antimony is best as the material of the reaction tube.

In preparing the tube it is best that the lead be melted at less than 350°C during preparation in order to avoid the formation of lead oxide. Using these materials and this procedure, the reaction tube becomes very anti-corrosive and can be used perfectly for more than two months.

I. INTRODUCTIONA. History of Project

In producing hydrogen peroxide by the hydrolysis of ammonium persulphate, the so-called pure lead tubes had been used previously in service as the reaction tube, but these tubes were corroded rapidly and damaged after a week. Therefore, we tried to make the reaction tube more anti-corrosive and obtained one which seemed to be more satisfactory.

These tubes are now in use in all the plants of this type in Japan. A flow sheet of a typical plant of this type is presented in Plate I(B)2.

B. Key Research Personnel Working on the Project

Chemical Engineering Lieutenant UETA
Chemical Engineering Sub-Lieutenant A. MOCHIZUKI

II. DETAILED DESCRIPTIONA. Test Procedures

The reaction tube is shown in Figure 1(B)2.

At first, the state of corrosion was observed microscopically in a pure lead tube which had been damaged. It was recognized that in the pure lead tubes, lead grains were very large and lead oxides were developed on the boundaries of lead grains; and that the corrosion only occurred in the lead oxide zones, or the boundaries of lead grains. (See Figure 2(B)2)

In order to avoid corrosion it is necessary that the eutectoid be compactly filled between lead grains and that lead oxide be eliminated.

From the above point of view, it was supposed that lead containing from 0.2% to 2.0% antimony is more anti-corrosive than pure lead. And also, from the metallurgical point of view, lead seems to combine with oxygen in the atmosphere at temperatures in excess of 350°C, and this lead oxide is readily corroded by the fluid.

From these points of view, it was supposed that lead tubes containing from 0.2% to 2.0% antimony and melted at less than 350°C during working up would be the best. (See Appendix). Therefore, various tubes were used in the plant of Edogawa Manufacturing Co., Ltd.

ENCLOSURE (B)_aB. Experimental Results

Lead tubes containing 0.2%-2.0% antimony were very good and could be used in service perfectly for more than two months.

These tubes were observed microscopically and the following observations were made.

The lead grains were very small and lead-antimony alloy filled up the space between lead grains compactly so that intercrystalline corrosion did not occur.

III. CONCLUSION

A. The best material for the reaction tube is lead containing 0.2-2.0% antimony.

B. The melting temperature of lead should be maintained at less than 350°C in order to avoid the oxidation of lead.

The reaction tube which was made under these conditions has a long life and was used in service perfectly more than two months.

The study was not complete, since proper percentage of Sb in the alloy has not been investigated.

APPENDIX

1. As shown in the above phase-diagram, Figure 5, lead begins to form a eutectoid with antimony at the point of 0.2% antimony.
2. At high temperatures, the greater the amount of antimony, the smaller the tensile strength of Pb-Sb alloy becomes, and at the same time the easier the alloy is oxidized.
3. When the amount of antimony is less than 0.2%, the tensile strength of the alloy is small, the alloy is easily oxidized, and intercrystalline corrosion occurs.
4. Thus it was assumed that 0.2-2.0% antimony lead alloys would be proper for this purpose.

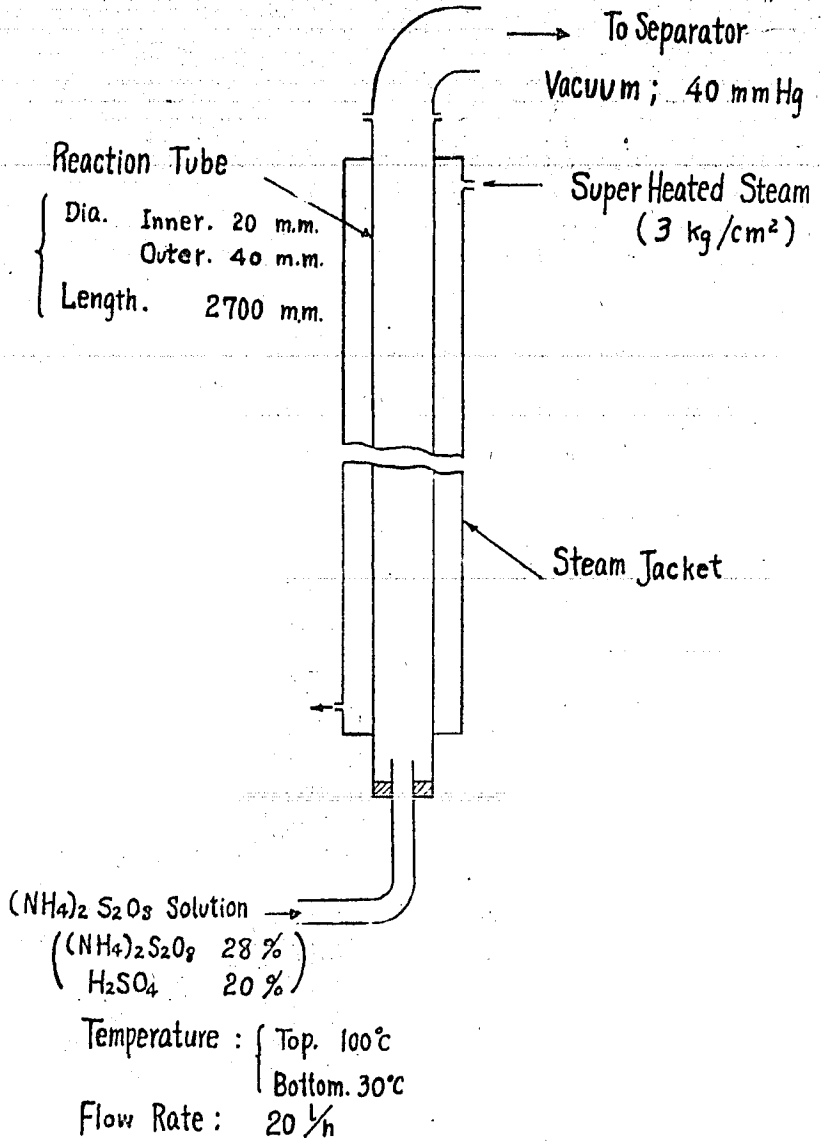


Figure 1 (B)2
REACTION TUBE

ENCLOSURE (B)2

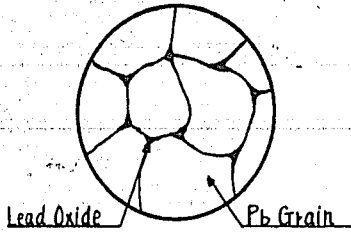


Figure 2 (B)2

PURE LEAD (0.016% Sb)

(x300)

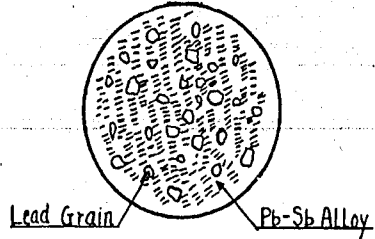


Figure 3 (B)2

LEAD CONTAINING 2% ANTIMONY

(x300)

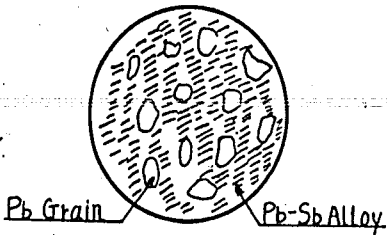


Figure 4 (B)2

LEAD CONTAINING 0.2% ANTIMONY

(x300)

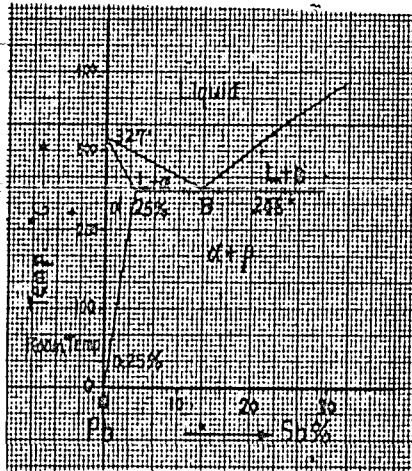
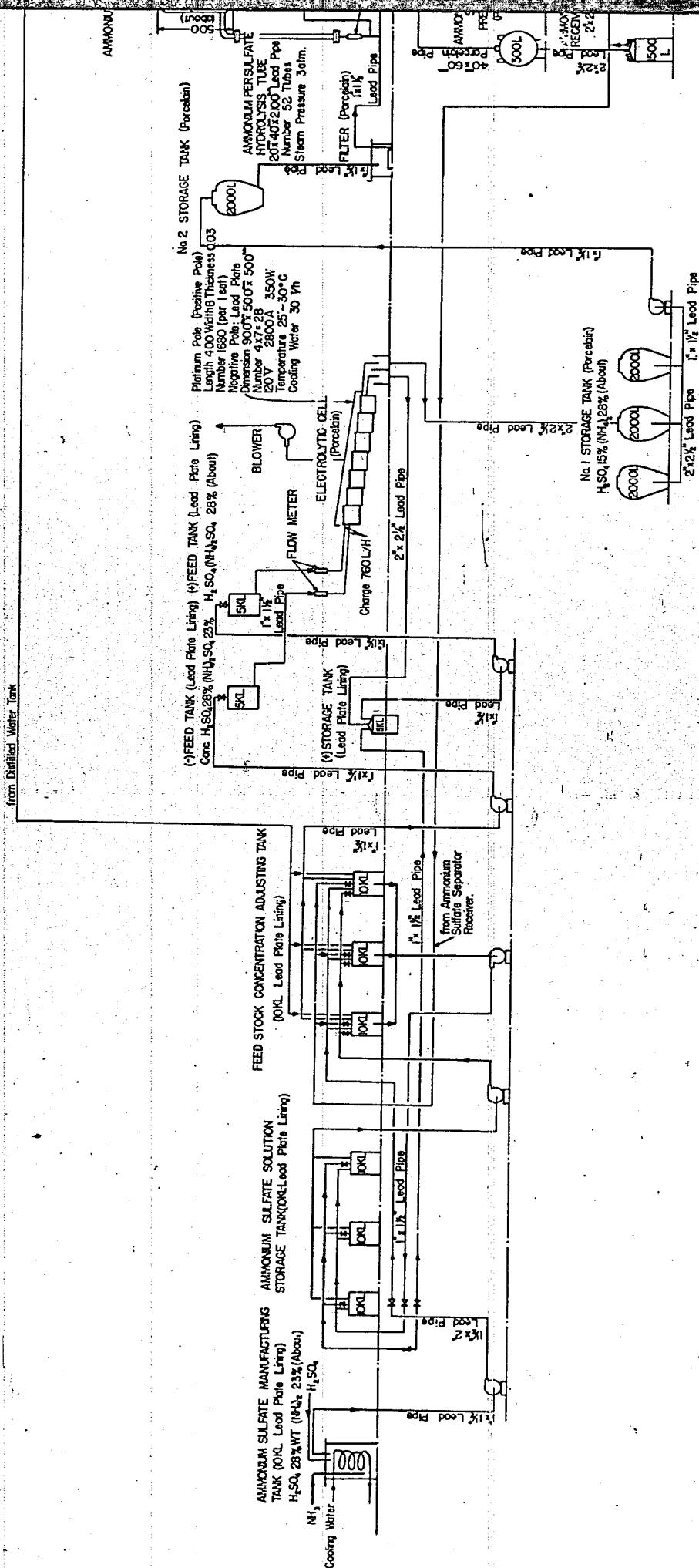


Figure 5 (B)2

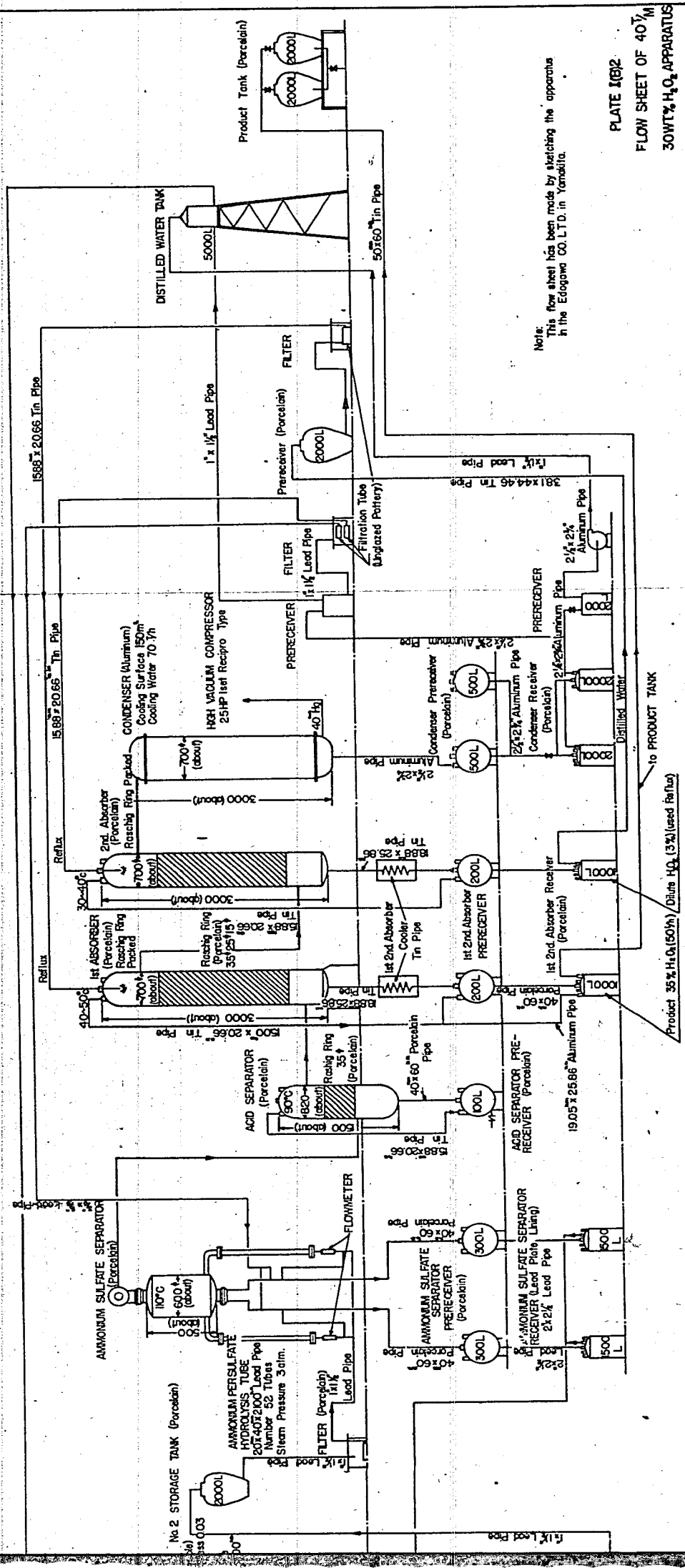
PHASE DIAGRAM



AMMONIUM

from Distilled Water Tank

from Ammonium Sulfate Separator Receiver



Note:
 This flow sheet has been made by sketching the apparatus
 in the Edogawa CO. LTD. in Yamoda.

PLATE 1(8)2
 FLOW SHEET OF 40%^M
 30WT% H₂O₂ APPARATUS