# THE USE OF INFRARED RADIATION FOR EVAPORATING WATER FROM PENICILLIN SOLUTIONS

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A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF SCIENCE IN CHEMICAL ENGINEERING

The University of Tennessee

May, 1949

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## SUMMARY

The application of infrared energy in concentrating solutions of penicillin by evaporation would permit the use of equipment that is much simpler than that now employed. Thermal lability of penicillin, however, must be considered; and the effects of infrared heating upon the antibiotic properties of penicillin were determined here. Also, the efficiency of evaporation and the temperature distribution in a solution exposed to infrared energy were investigated.

It was found that no temperature gradient measurable by ordinary thermometers is maintained in a liquid heated from above by infrared energy. After an induction period, the length of which depends upon the quantity of liquid being heated, the temperature throughout the body approached and maintained a constant value.

The efficiciency of evaporation (the percentage ratio of heat used in evaporation to total heat input to the system) was found to vary linearly from 23 per cent at a temperature of evaporation of 43° C. to 39 per cent at a temperature of 72° C.

A solution of penicillin having an initial concentration of approximately four units (one unit equals 0.6 microgram) per milliliter was exposed to infrared heat for three hours, attaining a maximum temperature of 55° C. and a

reduction in volume of one-third. The decrease in the potency of the penicillin amounted to 67 per cent.

It is recommended that further work on this subject include an investigation of the feasibility of using lower evaporation temperatures and thinner layers of solution in order to inhibit destruction of the drug. The use of infrared energy may also find application in the concentration of other heat sensitive materials similar to penicillin.

# THE USE OF INFRARED RADIATION FOR EVAPORATING WATER FROM PENICILLIN SOLUTIONS

## INTRODUCTION

In the production of penicillin and other biological materials, a problem is encountered in the concentration of the substances because of their thermal lability; that is, heat sensitivity is quite critical, and exposure to temperatures above a definite point results in destruction or alteration of valuable properties of the material. When the production of these sensitive materials is accomplished by fermentation, concentration usually entails the removal of large quantities of water from a solution, the product being dried for packaging. The process of concentration used at present on an industrial scale involves sublimation of water from frozen solutions under very low pressures; and it is apparent that equipment and operating techniques are complicated by the necessity of maintaining high vacuums and low temperatures.

The use of infrared energy for evaporation of water from solutions of antibiotics has been practiced in the bacteriology laboratories of the University of Tennessee; and the favorable results of this use prompted a survey of the possibility of adapting infrared energy to evaporation on a larger scale.

It was theorized at the outset that infrared rays are likely to be absorbed in the surface layer of a liquid when such rays are projected downward upon the liquid and, if so, that the exposure of a solute to heat arising from the rays would be lessened because of the tendency of a solute to have a lower concentration in the surface layer of a solution than in the body of the solution.[1] As a consequence of this speculation, it was decided to determine if a temperature gradient is maintained from surface to bottom layers of a solution exposed to infrared energy.

Another primary consideration was the determination of the efficiency of evaporation which could be obtained, using infrared radiation; i. e., the ratio of the amount of heat actually used in evaporating water to the amount of heat, as power, utilized in the infrared generator. In a practical application, such efficiencies would necessarily affect the economics of processing solutions.

As a natural consequence of the investigations previously mentioned, it was deemed worthwhile to carry out the evaporation of a solution of penicillin, noting the alteration of antibiotic strength resulting from exposure to infrared radiation for a given length of time and at a given, stable temperature.

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[1]Gibbs, Transactions of the Connecticut Academy, 3, 439 (1876).

## MATERIALS

In determining the evaporation efficiencies using infrared rays, distilled water was used, and was obtained from the still operated continually in the Chemical Engineering Department.

The penicillin used was the potassium salt, in the amount of 30.2 milligrams, containing 1606 units (one unit equals 0.6 microgram) per milligram and obtained from the Schenley Distilling Company. This amount of penicillin was put into solution in 48.2 milliliters of penicillin buffer solution obtained from the Bacteriology Department to give a mixture containing approximately 1000 units per milliliter. Using 249 milliliters of buffer and one milliliter of the initial solution, a solution of penicillin containing approximately four units per milliliter was obtained and used for the experimental evaporation.

Determination of the amount of destruction of penicillin by evaporation was effected by the use of a culture of the microorganism Staphylococcus aureus. This culture was obtained from the Bacteriology Department, and assay plates were made up from sterile nutrient agar as described in the section on procedure.

## EQUIPMENT

A schematic diagram of the equipment is shown in Figure 1.

The infrared source was a standard screw-base Westinghouse Infrared Lamp of 200 watt capacity and having a clear bulb. This lamp was mounted in a porcelain socket and was fitted with a gold-plated metal reflector. An energy distribution curve for this type of lamp is shown in Figure 5 of the appendix.

In order to minimize heat losses, the experiment was carried out in an insulated box made of plywood, the walls of which were filled with glass wool. The box had a lid, insulated as the walls, and fitting tightly by means of a gasket around the inside of the box. The bottom of the box was plywood, and the box was set on insulating board (Celotex). The infrared lamp was mounted on the inner surface of the lid of the box and was supplied with current through a cable passing into the box by way of a caulked hole in the lid. A $\frac{1}{4}$-inch copper pipe was inserted through one end wall of the box and a $\frac{1}{2}$-inch copper pipe was placed through the wall of the other end so that a stream of air could be used to sweep vapor out of the system. Mercury thermometers (0-100° C.) were placed in each of these pipes adjacent to the exterior walls of the box in order to measure inlet and exit air temperatures.

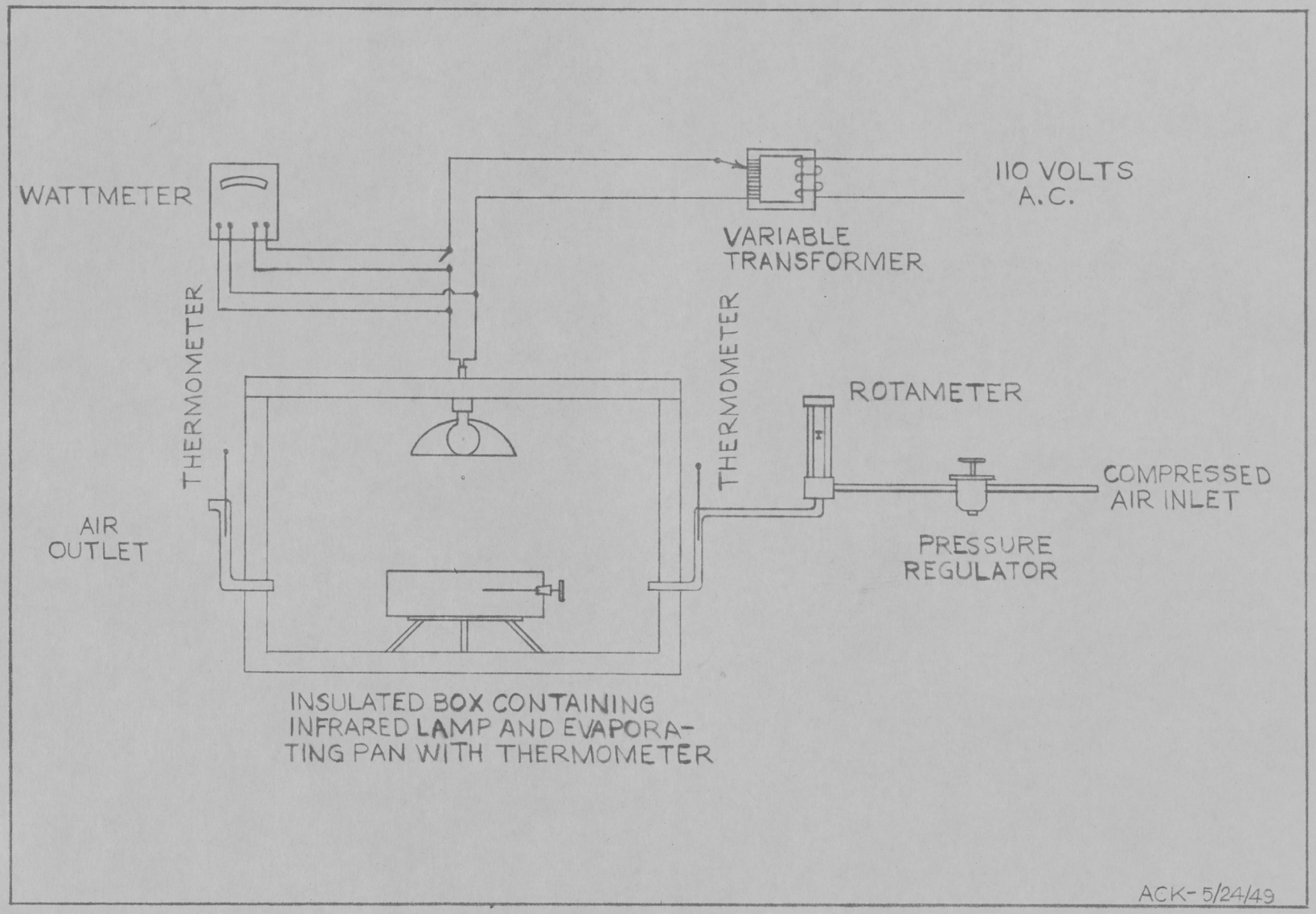



FIGURE 1
INFRARED EVAPORATION EQUIPMENT

A metal pan approximately 5 inches deep and 12 inches in diameter was made by cutting off a paint container and was used for the preliminary measurements of evaporation efficiency. Through a hole in the side of this pan, a Weston metal tube thermometer (0-100° C.) was inserted for measurement of water temperatures. For the evaporation of penicillin, a Pyrex glass dish approximately 4 inches in diameter and 3 inches deep was used.

Measurement of the air flow through the insulated box was effected by use of an oxygen rotameter made by the Linde Air Products Company. This meter was calibrated for measurement of air flow rates, using a Precision Scientific Company wet test gas meter; and the calibration curve appears in Figure 3 in the appendix.

Compressed air was used to sweep out the water vapor, and a pressure regulator manufactured by the National Cylinder Gas Company was employed to maintain constant air pressure and rates of flow.

The power input to the lamp was measured by means of a wattmeter, and voltage control was effected by means of a variable transformer manufactured by the Superior Electric Company.

## PROCEDURE

At the beginning of the investigation of the use of infrared radiation for evaporation, it was not known if a temperature gradient is set up in the body of a solution heated by the radiation. In order to establish the definite existence or absence of such a gradient, runs were made in which distilled water was evaporated by the infrared rays while temperatures were read continually at the liquid surface and at points one to two inches beneath the surface. In order to obtain the temperature of the surface liquid, a Weston metal tube thermometer was inserted by means of a rubber stopper into a hole in the side of the pan containing the water. The liquid level was maintained at the same level as the protruding thermometer, and an ordinary glass thermometer was used to measure temperatures at points well beneath the surface. Of course, some time was required for the attainment of an equilibrium, the result of which will be discussed later in the report.

The efficiency of the energy source as a means of evaporating water was next determined for various temperatures in the range near which penicillin is destroyed by prolonged exposure to heat. The evaporating pan was filled with water and placed in the insulated box beneath the lamp, the lid was replaced on the box, and the lamp was energized by adjusting the power input by means of the variable trans-

former. The rate of flow of air through the box was set at 8.7 grams per minute, and the temperature of the water was allowed to reach an equilibrium value as indicated by the thermometer inserted through the side of the pan. When equilibrium was established, the pan was removed, weighed as quickly as possible, and returned to the box, whereupon a timed run was begun. The power input and rate of flow of air were regulated continually to maintain constant conditions; and the following quantities were recorded: Time of run; initial and final weights of pan plus water; power input to the lamp; temperature at which evaporation occurred; air flow rate; and entrance and exit air temperatures. The efficiency of the evaporation, defined here as the ratio of the amount of heat utilized as latent heat of vaporization to the heat equivalent of the power input to the lamp, was then calculated.

Using different values of power input to the lamp and keeping the air flow rate constant, a series of evaporation efficiencies were determined for the resultant various evaporation temperatures. From these data, a curve was plotted which relates evaporation temperature to evaporation efficiency as defined in connection with this investigation.

The third phase of the problem was concerned with the concentration of a solution of penicillin in which the initial concentration was approximately four units per milliliter. This operation was carried out, using a Pyrex bowl instead of the metal pan used previously. The voltage

transformer was set to give a maximum temperature of the solution of 55° C. and the air flow rate was maintained constant as before at 8.7 grams per minute. One hundred milliliters of the solution were placed under the lamp, after preheating the box, and allowed to remain for three hours. At the end of this period, the volume of solution remaining was measured and its antibiotic strength was determined.

The effect of the exposure to heat during the evaporation was measured by comparison with a sample of the original solution which was refrigerated during the test run. For this assay, cultures of the microorganism Staphylococcus aureus were used as follows: The organism was allowed to grow at 37° C. for 24 hours in about 10 milliliters of nutrient broth and was then transferred to 100 milliliters of sterile nutrient agar which was cooled to about 40° C. immediately after autoclaving 20 minutes under 18 pounds steam pressure. This agar was then poured into sterile Petri dishes to a depth of about 4 mm., and the plates were kept in a refrigerator until required. In testing the strength of the solutions, standard absorbent paper discs were saturated with each solution, known and unknown, placed on the agar surface in alternating sequence, four to the plate, and incubated about 18 hours at 37° C. The antibiotic strength of each solution was measured by the

diameter of the zone of inhibition of growth of the micro-organism, making use of a curve relating the diameter of inhibition zone to strength of solution, measured in units of penicillin per milliliter (One unit of penicillin equals 0.6 microgram). In order that the zones could be measured, it was necessary to dilute the solutions 1:4 before the assay was made.

## RESULTS

The conclusion reached following the runs designed to determine the presence or absence of a temperature gradient in a body of liquid exposed to infrared radiation was that no appreciable gradient is maintained after an induction period which brings the system to equilibrium.

A plot of evaporation efficiency versus the temperature at which evaporation occurred is shown in Figure 2. The experimental data from which the efficiency curve was plotted appears in Table I of the appendix.

Using the penicillin assay curve prepared by the Bacteriology Department and reproduced in Figure 4 of the appendix, it was found that destruction of 67 per cent of the initial amount of penicillin occurred in the three-hour exposure to infrared heating, where one-third of the initial volume of the solution was evaporated at $55^{\circ}$ C.

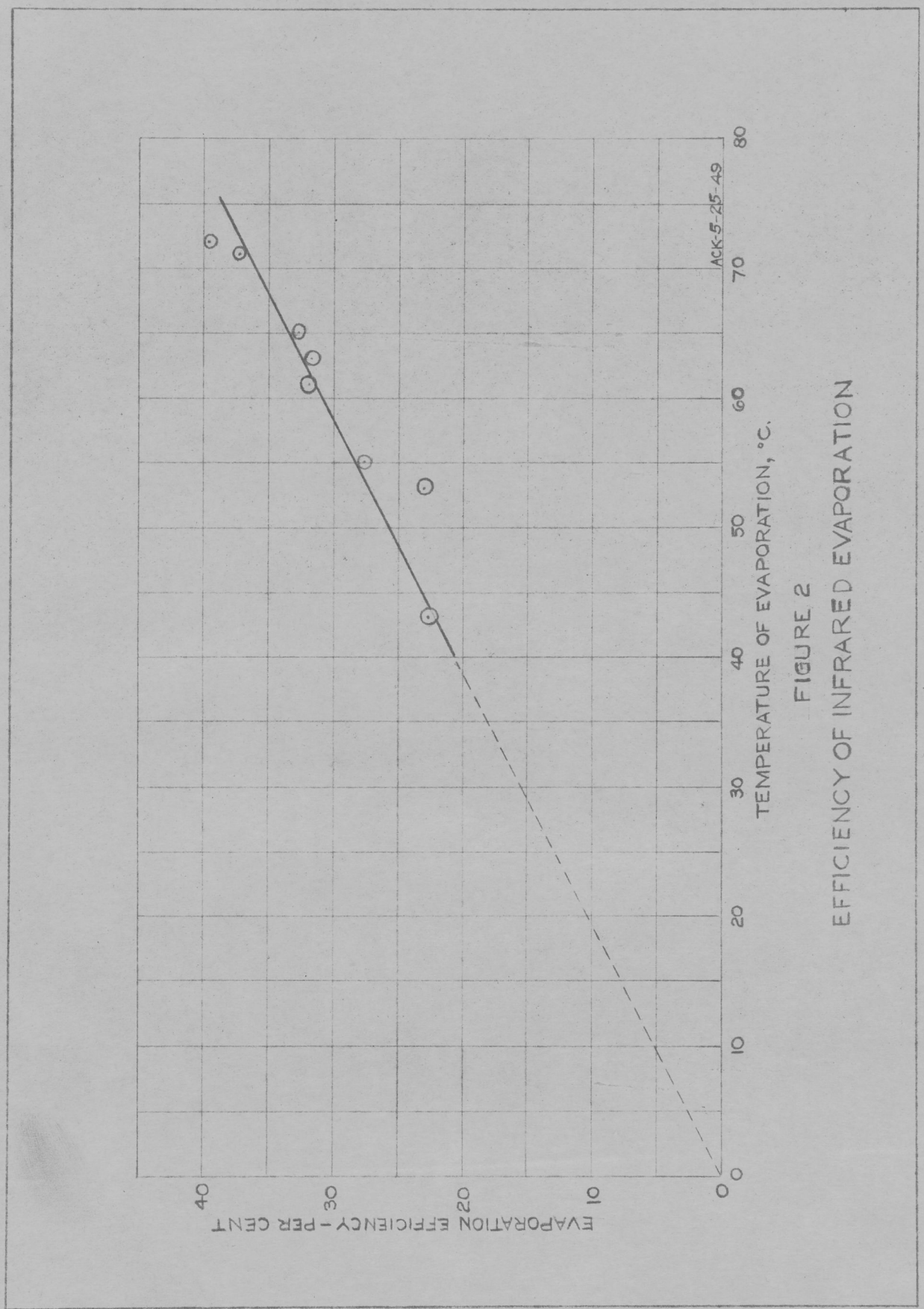



FIGURE 2

EFFICIENCY OF INFRARED EVAPORATION

## DISCUSSION OF RESULTS

The conclusion that there is no appreciable temperature gradient maintained in a bath exposed to infrared radiation is not surprising in view of the conditions which prevail in such a situation. Although the rays of infrared energy may have low penetrating power and fail to reach far below the liquid surface, the heat effect cannot be confined to surface layers because of the thermal conductivity of the solution. In this case, water being used, the thermal conductivity at the temperatures involved (40-70° C.) is upward of $1.5 \times 10^{-3}$ calories/ second centimeter °C.[1], which is close to that of an ordinary brick. Whereas a solution heated from below reaches a constant temperature because of the mechanisms of conduction and convection, presumably one heated from above would attain equilibrium more slowly through conduction, convection depending on changing liquid densities and being ineffective where the heated layers already overlie cooler layers.

The variation of evaporation efficiency with the temperature of vaporization is linear, as shown in Figure 2, and extrapolates to zero efficiency at 0° C., where no heat would be added and no vaporization would occur.

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[1] Lange, Handbook of Chemistry, Sixth Edition, Handbook Publishers, Incorporated, Sandusky, Ohio, 1946. 1503, 1505.

The efficiencies obtained here, varying from 25 to 40 per cent are in close agreement with those found in applications of infrared radiation to water evaporation.[1] Factors which lower the efficiency are: (1) Infrared lamps are not 100 per cent efficient as infrared generators; (2) Reflectors do not redirect all the infrared energy; (3) Not all the energy radiated by the lamp and reflector is accepted by the material exposed to radiation; (4) Energy is lost due to reradiation, since any heated object or material is a secondary infrared generator. Infrared lamp and reflector units are found to be roughly 65 per cent efficient in the generation of infrared rays.[2]

The destruction of penicillin resulting from thermal exposure during evaporation indicates the feasibility of lowering evaporation temperatures and shortening the heating period. This could be done by conducting the evaporation with a thinner film of solution than was used here.

The investigation as carried out constitutes a good foundation for further research on the subject of infrared evaporation as applied to biological and other heat sensitive solutions. It is recommended that additional data be taken using more refined methods of measurement and control than were possible under the present circumstances.

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[1]Hall, Industrial Applications of Infrared, McGraw-Hill, New York, 1947. 58.

[2]Hall, op. cit., p. 58.

For instance, liquid temperatures should be measured by thermocouples or resistance thermometers, which would permit a more accurate observation of temperature distribution in the liquid. An integral weighing system would be desirable in the insulated compartment, which should itself have better insulation and possibly a lining of aluminum foil. Such a weighing system would obviate the opening of the box during evaporations in order to weigh the water, which operation allows an incalculable loss of heat. Measurement by means of a radiation meter of the actual radiation emitted by a given generating unit would be desirable in order to arrive at reliable power requirements. These are some of the more important refinements of technique which might prove helpful in future investigations of like nature.

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ACKNOWLEDGMENT

The author wishes to express his appreciation to Mr. A. L. Pollard for his instructional assistance and for the loan of equipment; to Messrs. Ben Thomas and Jim Eubanks for their aid in assembling equipment; and to various members of the Chemical Engineering Department staff for their advice on matters of technique.

# APPENDIX

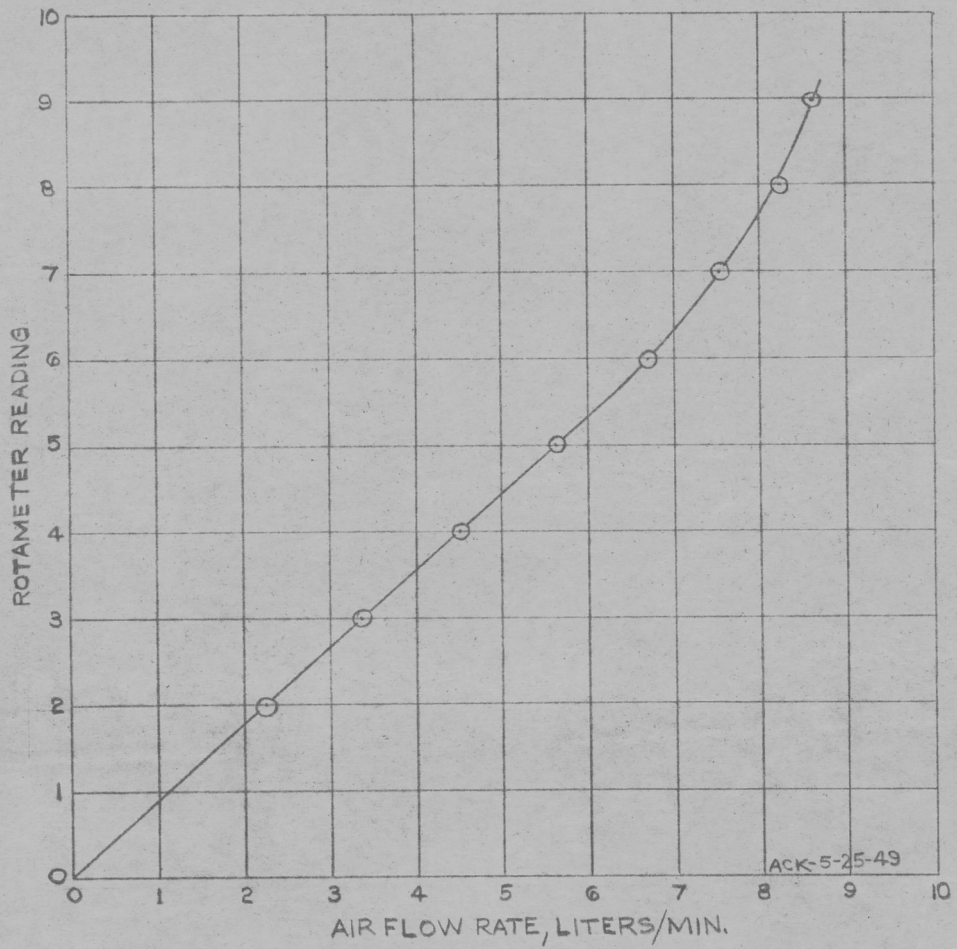



FIGURE 3

ROTAMETER CALIBRATION

TABLE I

DATA FOR INFRARED EVAPORATION EFFICIENCY

| Evaporation Temperature, Degrees C. | Power Input to Lamp, Calories | Length of Evaporation Period, Hours | Weight of Water Evaporated, Grams | Heat Required to Evaporate Water Removed, Calories | Efficiency of Evaporation, Per Cent |
|---|---|---|---|---|---|
| 43 | 43,000 | 1.0 | 17 | 9,740 | 22.6 |
| 53 | 120,500 | 1.0 | 49 | 27,800 | 23.1 |
| 55 | 86,000 | 1.0 | 42 | 23,800 | 27.7 |
| 61 | 155,000 | 1.5 | 88 | 49,500 | 31.9 |
| 63 | 129,000 | 1.0 | 73 | 41,000 | 31.8 |
| 65 | 129,000 | 1.0 | 76 | 42,400 | 32.8 |
| 71 | 179,000 | 1.0 | 120 | 66,900 | 37.4 |
| 72 | 258,000 | 1.5 | 184 | 102,300 | 39.7 |

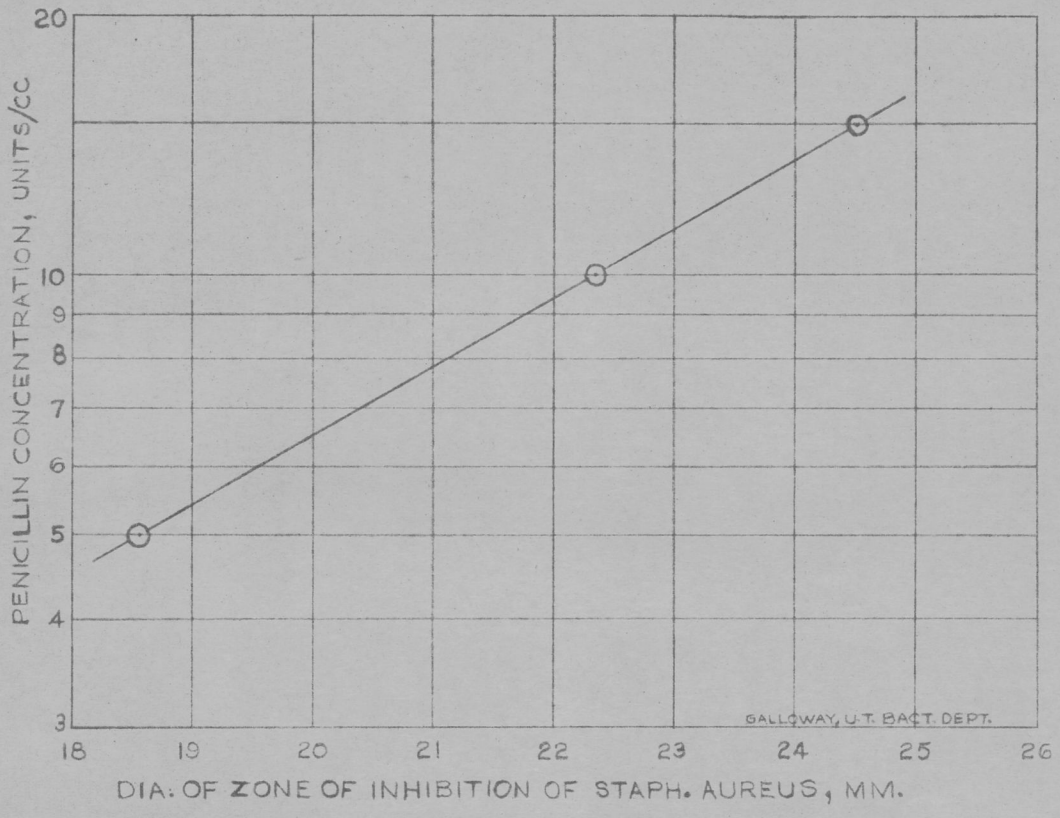



FIGURE 4

PENICILLIN ASSAY CURVE

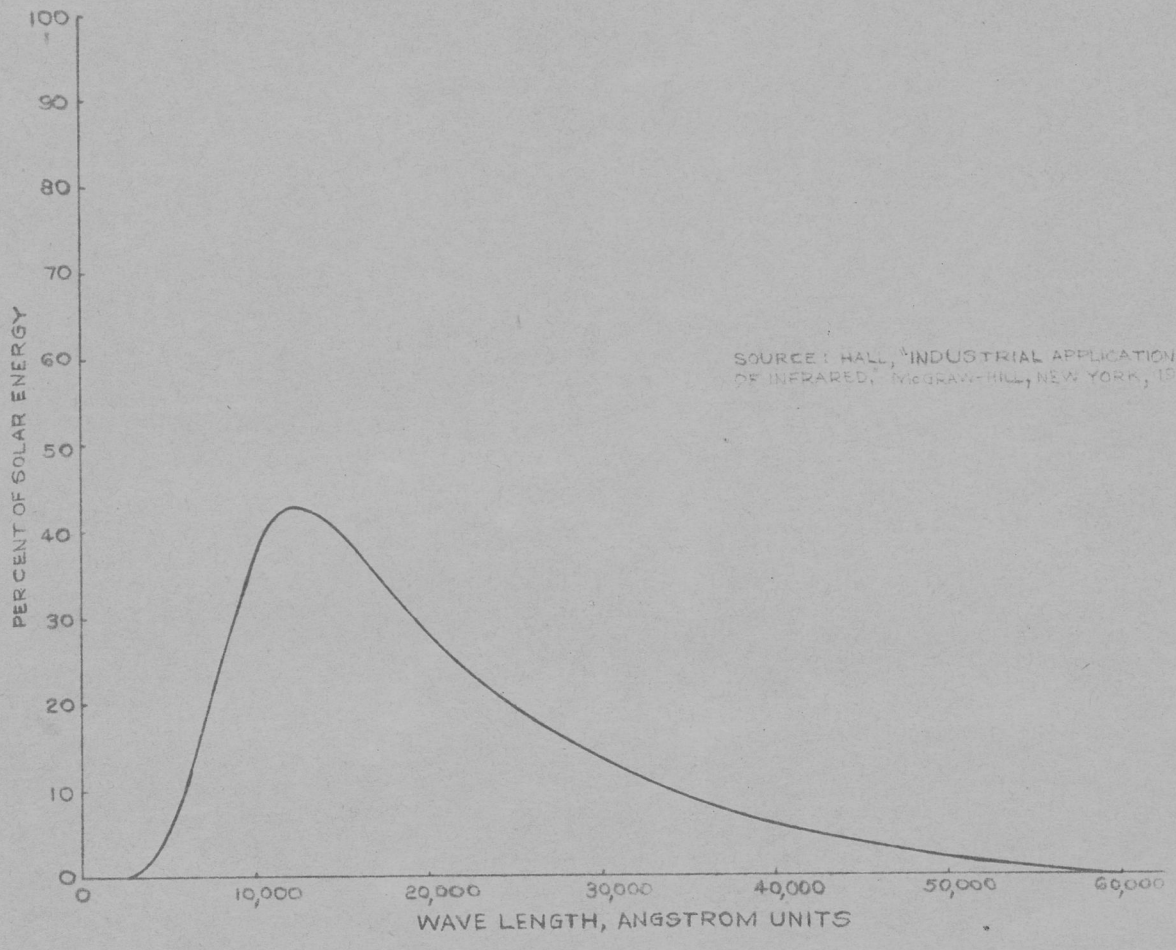



FIGURE 5

ENERGY DISTRIBUTION OF INFRARED LAMPS, BASED ON SOLAR ENERGY

## APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinions expressed, or conclusions drawn therein, but approves the thesis only for the purpose for which it is submitted.

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