Chapter V

EXPERIMENTAL CONSIDERATION

5.1 Calibration of Flow Meter

There were three flow meters to be calibrated: two orifice-meters and one rotameter.

5.1.1 Calibration of orifice meters

Two orifice-meters were used to indicate the flow rates of hot water and cold water. When water flows through an orifice-meter, a pressure drop is developed for a particular flow rate. This pressure drop is generally indicated by a mercury manometer.

Calibration of each of the orifice meters was carried out by varying the rate of water flowing through the meter. Each flow rate was calculated by recording the time required to collect a certain amount of water flowing through the meter. The corresponding pressure drop for each flow rate was also measured from the attached manometer, and it was recorded. The calibration of each meter were plotted as flow rate in kg/min versus manometer readings in cm.Hg, as shown in Figures 7 and 8.

5.1.2 Calibration of rotameter

A rotameter was used to indicate the flow rate of fruit

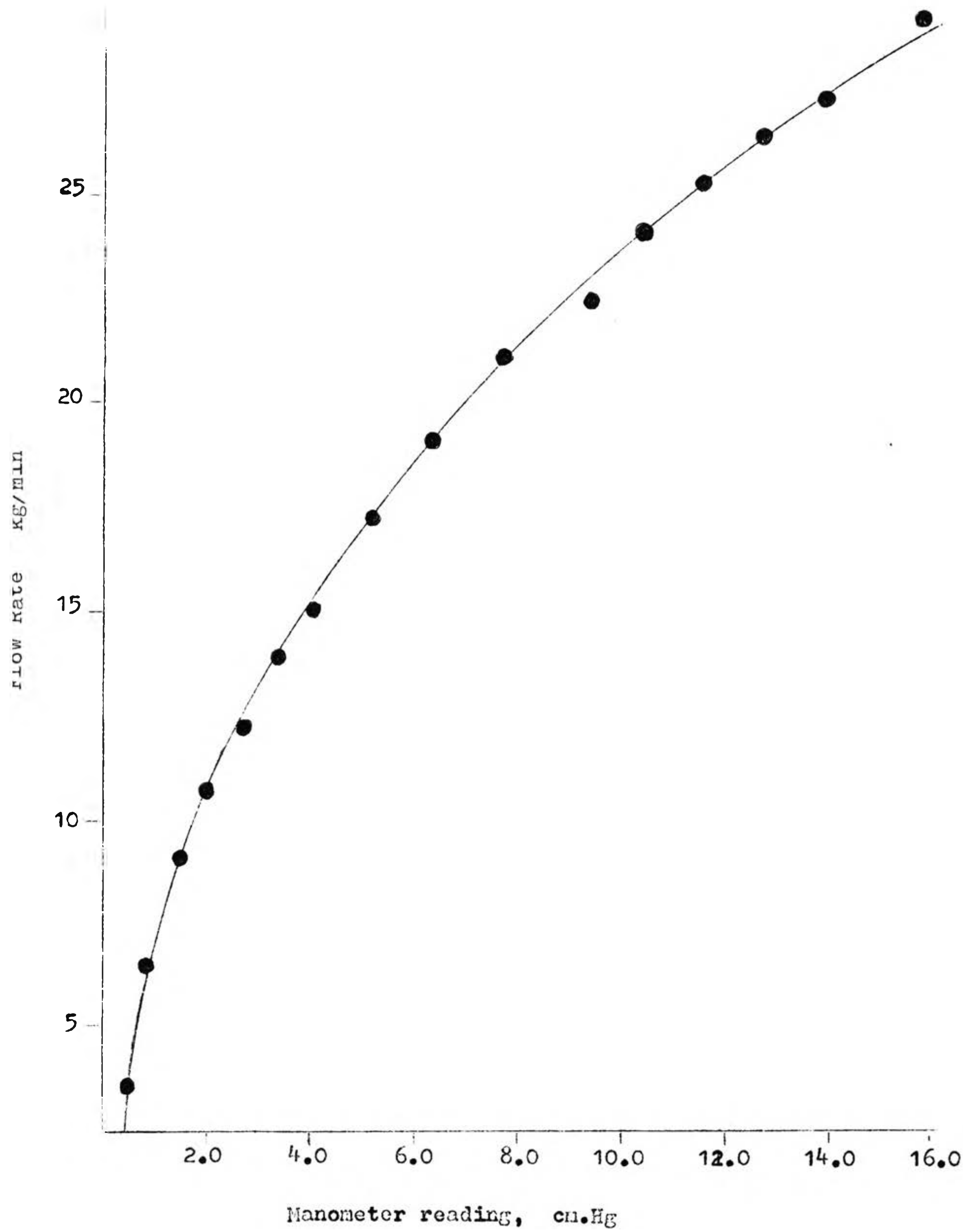

Figure 7. Calibration of Hot-water Orifice-meter

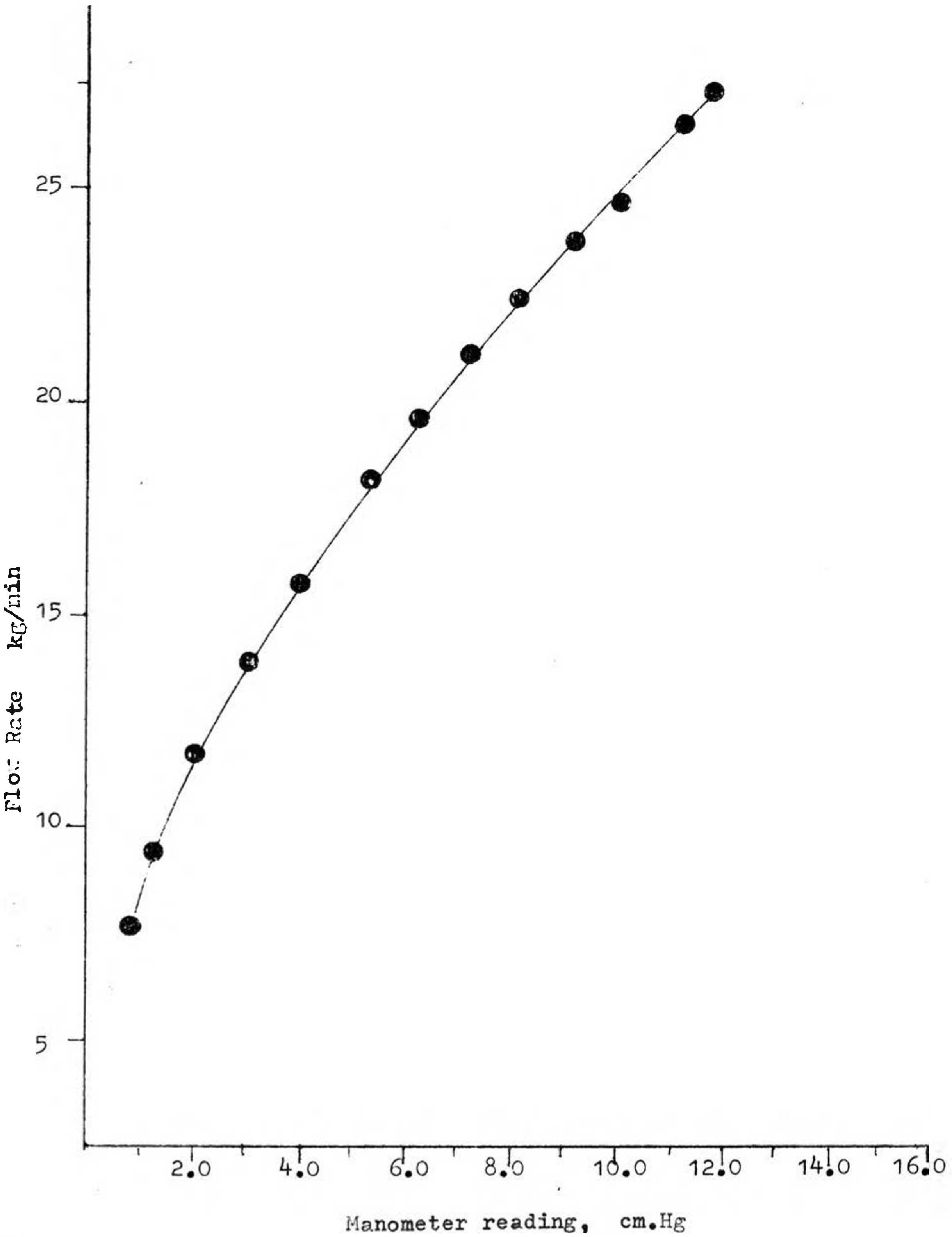

Figure 8. Calibration of Cold-water Orifice-meter

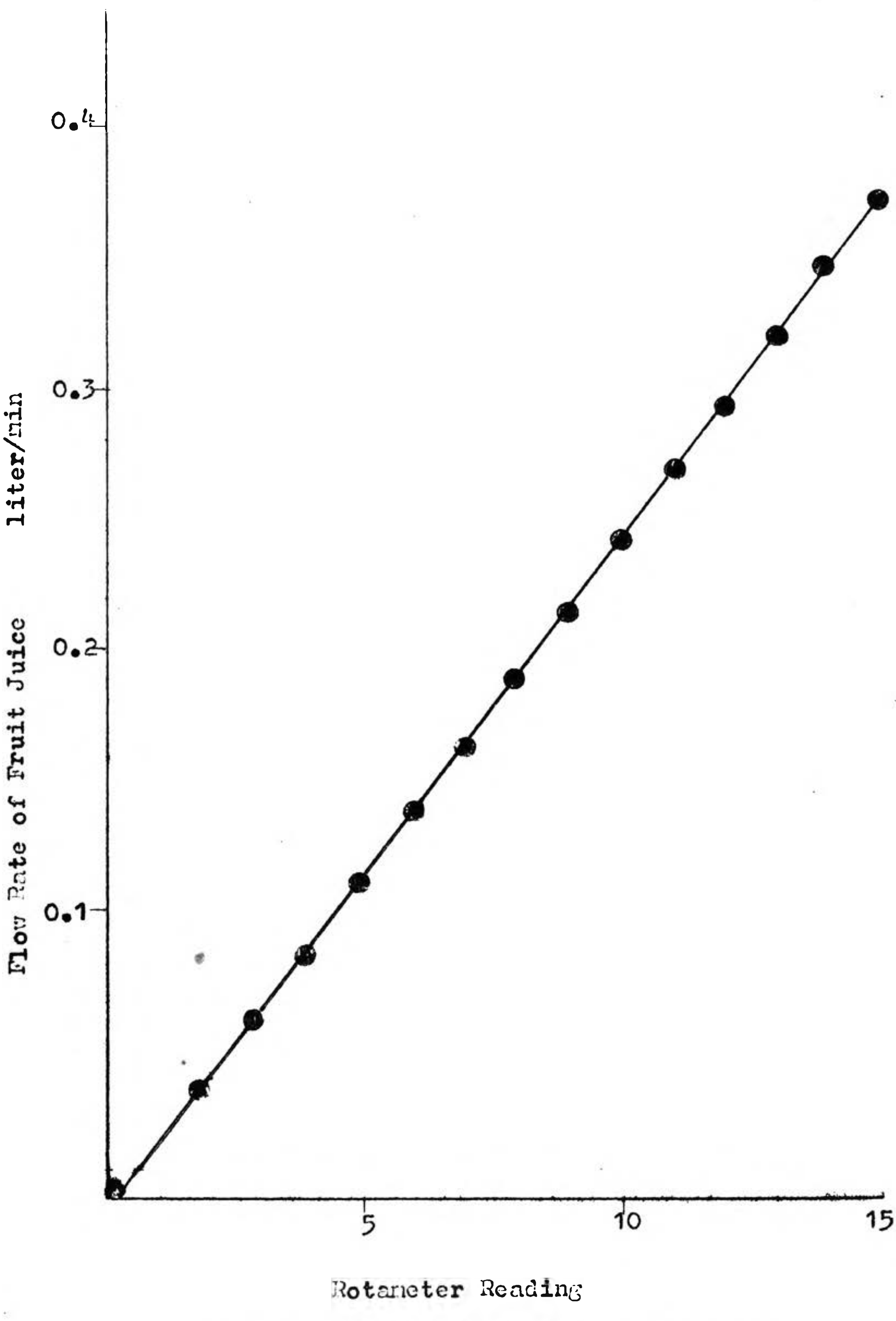

Figure 9. Calibration of Feed Rotameter

juice entering the inclined-film evaporator. The meter has an attached scale, of 15 divisions, each divided into 10 subdivisions.

The rotameter was calibrated by varying the rate of fruit juice flowing through the meter. The flow rates were calculated by recording the amount of fruit juice flowing through the meter collected in one minute. The corresponding meter reading from the attached scale for each flow rate was also read and recorded. The calibration data were plotted as flow rate in liter/min versus meter reading, as shown in Figure 9.

5.2 Experimental Variables and Experimental Runs

The present experiments would be carried out using juice from Citrus reticulata Blanco.

5.2.1 Experimental variables

The process variables which would be studied were:

(a) Independent variables

(i) the feed rate of fruit juice. Six feed rates would be used, and they were corresponding to the feed rotameter readings of 10, 11, 12, 13, 14, and 15.

(ii) the temperatures of hot water. Seven mean temperature of hot water inlet would be used, they are 84.6,89.2,87.8,88.8,87.7,85.7 and 85.9°C

(b) Dependent variables which would be measured were:

(i) the concentration of juice before and after processing

(ii) pH of juice before and after processing

(iii) acidity, expressed as citric acid, of juice before and after processing

(iv) vitamin C content of juice before and after processing

(v) pressure of steam in the heat exchanger

(vi) temperatures of inlet steam and outlet condensate of the hot-water heater

(vii) flow rate of hot water

(viii) temperatures of cold water inlet and outlet in both condensers

(ix) flow rate of cold water

(x) vacuum in the evaporating chamber

(xi) temperature of the evaporating juice

(xii) temperatures of condensate inlet and outlet in both condensers.

5.2.2 Experimental runs

The experimental runs which were planned to be performed were:

1. Test run of the equipment: Sugar solution of concentration 9-11% w/w would be used with varied feed rate and varied temperature of hot water in order to search for the proper range of feed rate and the proper steam pressure.

2. Experimental runs: Experiments were planned to be performed using orange juice, and there were seven runs as followed.

Run no	Feed rotameter	Temp.of Hot water °C
1	10	89
2	11	90.5
3	12	89
4	13	91.9
5	14	89
6	15	90.5
7	11	87

5.3 Experimental Procedure

The procedure used in the present experiments consisted of several subsequent steps as shown in Figure 10.

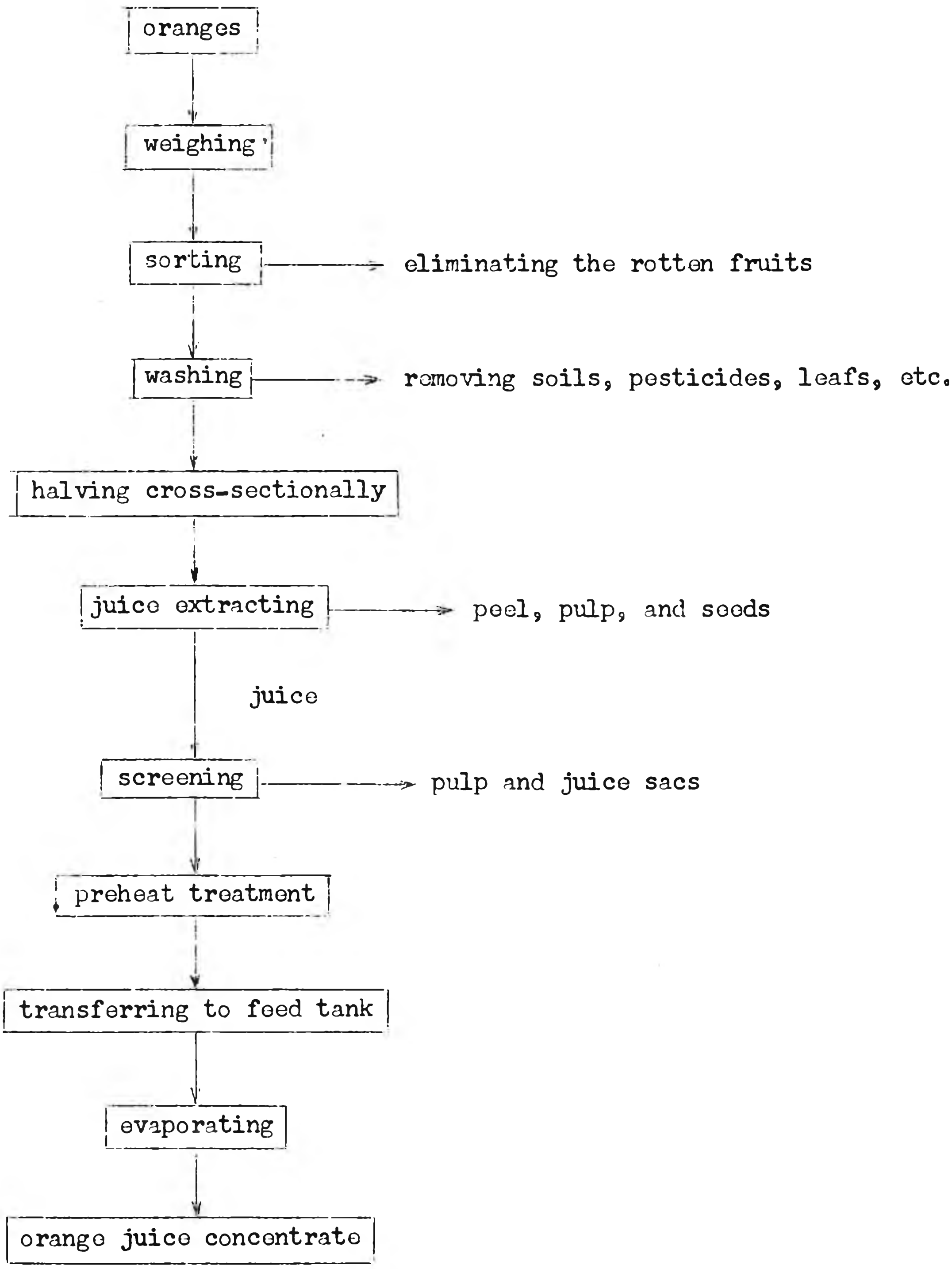

Figure 10. Block diagram of experimental procedure

Fifty kilograms of Citrus reticulata Blanco orange was received from a distributor and used for each experimental run. After weighing, sorting, washing, halving, juice was extracted from the fruit on a Kenwood Juice Extractor Model KNM 6, screened on a plastic screener to remove seeds and coarse particles even some pulp. It was followed by screening with double fold of screening cloth to remove juice sac. The screened juice, weighed about 30% of the original fruits, was passed through a double coil made of stainless steel of 0.92 cm diameter and 6 meter long which was keeping in boiling water all the time. At the end of this coil, the heated juice of a temperature between 70-80°C was stored in a glass container which was tightly closed. The juice was sucked through the coil by applying some vacuum to the glass container. This was also deaeration and pasteurization.

By connecting a PVC tubing from the feed tank (1) (see Figure 11) to the glass container and applying some vacuum to the former, the juice was then sucked to the feed tank. The temperature and concentration of the juice were measured. The juice was fed into the evaporating chamber (2), the feed rate was controlled by a feed valve (3) and was determined by a rotameter (4). It flowed through a distributor (5) onto the heated plate (6) and overflowed from a wier (7) as a thin film. The plate, beneath of which situated a long rectangular chamber for circulated hot water, was wetted before with some juice while being heated to make the film flow smoothly. The hot water was circulated by a 0.35 H.P. centrifugal pump (8) through

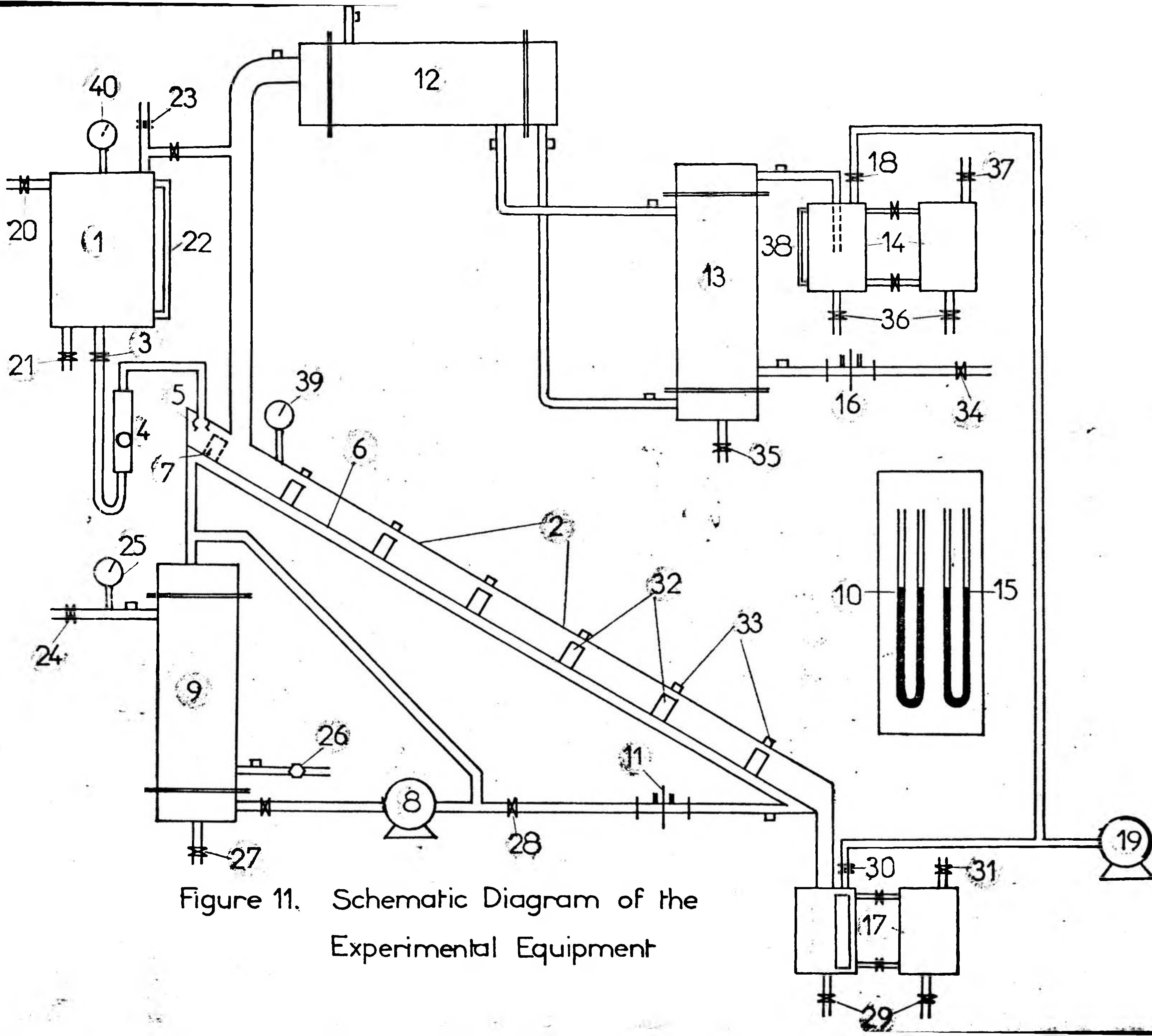

Figure 11. Schematic Diagram of the Experimental Equipment

1. Feed tank
2. Evaporating chamber
3. Feed valve
4. Rotameter
5. Distributor
6. Inclined heated plate
7. Wier
8. Centrifugal pump
9. Hot water heater
10. Manometer for hot water
11. Orifice-meter for hot water
12.,13. Condensers
14. Condensate tanks
15. Manometer for cold water
16. Orifice-meter for cold water
17. Concentrated-juice tanks or receiver tanks
18. Vacuum valve
19. Vacuum pump
20. Feed inlet valve
21. Drain valve
22. Glass tube
23. Vent valve
24. Steam inlet
25. Steam pressure gauge
26. Steam trap
27. Hot water inlet/draining
28. Hot-water controller
29. Concentrated-juice draining valves
30. Line to vacuum pumps
31. Vent valve
32. Glass windows
33. Thermometer ports
34. Cooling water inlet valve
35. Drain valve
36. Condensate draining valves
37. Vent valve
38. Glass tube
39. Vacuum gauge
40. Vacuum gauge

a shell and tube heat exchanger (9), which was heated by steam on the shell side. The temperature of the plate, which was assumed to be the same as the temperature of the hot water, was controlled by the flow rate of hot water indicated by the manometer (10) of an orifice meter (11)

In the evaporating section (2), the vacuum was applied at the beginning of the run to reduce the boiling point of the juice and to force the juice flow through the rotameter (4). Then evaporation took place, the vapors passed through a horizontal condenser (12) in tube side and then a vertical one (13) in tube side too, to a condensate tank (14). The cooling water passed through the shell side of both condensers, and the flow rate was measured by the manometer reading (15) of an orifice meter (16).

The concentrating juice flowed downward under gravity along the inclined plate to a receiver tank (17). During the evaporation the followings were recorded every five minutes.

1. feed rotameter reading
2. pressure of steam entered the heat exchanger
3. temperatures of steam both inlet and outlet
4. temperatures of hot water both inlet and outlet
5. flow rate of hot water
6. temperatures of cold water inlet and outlet in both condensers
7. flow rate of cold water
8. vacuum gauge of the evaporating chamber

9. temperature of the evaporating juice

10. temperature of vapor in both condensers.

As the fruit juice in the feed tank had been used up.,the experimental run was terminated. To stop the run the steam inlet valve, the hot-water pump, then the vacuum valve(18) and vacuum pump(19) were turned off. The weight, temperature and concentration of the concentrate-juice, and the weight of condensate were recorded. The time consumed for the experimental run was also recorded.

5.4 Experimental Analysis

Physical and chemical analysis of orange juice before and after processing were carried out as follow:

Physical analysis

1. Concentration, as expressed in Brix and being assumed as percent total soluble solid or solid content, was measured by a hand refractometer of ATAGO,Japan No.124317.

2. pH was measured by pH meter,No.7010 of Electronic Instruments Ltd., which was calibrated using prepared buffer solution of pH 4.7.

Chemical analysis

1. Acidity: Acidity of orange juice was determined by using

Indicator Method.[1] Orange juice of 10ml.for fresh juice,or 10 ml. diluted to 20 ml.and used 10 ml.for concentrate-juice, was titrated with standardised 0.1N NaOH solution using 0.3 ml.phenolphthalein as indicator. From ml.of NaOH used,the quivalent of acid was then obtained and further calculated as gm citric acid.

2. Vitamin C: The vitamin C content was determined using Iodine Titration Method.[2] Sample of 10 ml.for fresh juice,or 25 ml. for concentrate-juice,was titrated rapidly with standardised 0.01 N Iodine solution[3] using 1% starch solution[4] as indicator. From ml. Iodine used,vitamin C content was calculated.

5.5 Experimental Observations

During the experiments,some difficulties were confronted and some observations were also noted.

1.Initially,the feed juice was introduced to the evaporating chamber by the distributor alone. This cause unsmoothed thin film since it flowed locally in disconnected streams like chanelling. Only at very high feed rates the plate was found to be completely covered by fluid film but of varying thickness. However such feed rates were too high for the present experiments. It was thought that either the distributor did not function well or the plate was not clean enough. The dirt made impossible smooth flow of the film.

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[1] William Horwitz,Official Methods of Analysis of the Association of Official Analytical Chemists(11thed. Wisconsin:George Banta Company,Inc.,1970),p.377.

[2] Morris M.Jacobs,Chemical Analysis of Foods and Food Products (New Jersey:D.Van Nostrand,Co.Inc.,1959),pp.724-727.

[3] Arther I.Vogel, Text-book of Quantitative Inorganic Analysis (3ded. G.B: Richard Clay,Ltd.,1961),pp.348,357.

[4] Ibid.p.347.

Thus, to solve the problem, the plate was cleaned and a wier, 1.27 cm. high and 0.157 cm. thick of stainless steel, was welded to the plate at the position close to the distributor. The juice then flowed through the distributor onto the plate and overflowed the weir. Smoother film was then produced. However, it was still necessary to have high feed rate at the beginning of the run to wet all the plate, and then the rate could be reduced to a desired value at which the smooth film still existed.

2. To test run the equipment, prior to concentrating orange juice, some experiments were performed using sugar solution of concentration 9-11% w/w measured by a hand refractometer and expressed in Brix. The solution was warmed to 70°C before introducing to flow in order that water would vaporize as it entered the evaporating chamber. The running of the film was found unsmooth along the plate even the overflow on the wier was adjusted. It might be possible that the plate itself was not smooth enough. Crystallisation of sugar was also found during the evaporation at some dried spots on the plate. The change in viscosity was noticeable. More sugar crystals were observed at the end of the plate rather than at the beginning. The boiling of the liquid disturbed the flow and some foaming occurred.

3. The flow of the feed was not constant at desired rate. Some bubbles developed in the feed line and pushed the float in the rotameter vigorously. The fluctuation seemed to increase as the head in the feed tank decreased. This also corresponded to

the vacuum in the evaporating chamber, as the liquid boiled the vacuum decreased. It was thought that that was due to either:

3.1 the suddenly change of pressure, from atmospheric in the feed tank to under vacuum in the evaporating chamber, thus, the warmed feed could boil in the line,

or 3.2 the leakage around the joint of the line which permitted air from the outside entered and formed air bubbles in the line,

or 3.3 the use of gate valve instead of a glove valve in the feed line, the former is not a good flow-rate regulator as the latter.

4. The fluctuation of rotameter reading of the feed and the fluctuation of vacuum gauge reading of the evaporating chamber seemed to have some relationship. As the vacuum increased the float of rotameter rose up, and as the vacuum decreased the float sank. It was apparent that when the feed was sucked into the evaporating vacuum chamber, the feed temperature was higher than the boiling point in the chamber. The vaporization immediately took place, the vapor pressure increased, so the vacuum dropped and this consequently caused the float to sink. As the feed rate decreased, less water was vaporized in the chamber, the vacuum consequently increased. This phenomena seemed to be a cyclic process.

5. To run the experiments, orange juice was used. It was found that filter press could not be applied to filter this juice, even the high pressure developed in the pump. It was the particle size of pulps and the formation of gel which resisted the flow

through the filter paper. Therefore plastic screen placed in the juice extractor and screening cloth were used instead of the filter press.

6. The temperature of evaporation was too high, this was unfavorable for aromatic substances in the concentrated-juice. The characteristics of fresh orange juice, greatly based on aromatic substances, after evaporating changed a lot, though the juice body and color were still appreciated. The flavor was found to be traped in the condensate and also in the vacuum oil used for vacuum pump. The leakage in the evaporating chamber was checked and rechecked in order to eliminate even the least leakages. The oil used in the vacuum pump was tried to obtain the most effective one. The maximum vacuum produced in the chamber was still too low. It was found literally that the temperature of evaporation which resulted the minimum loss of flavor is 21 °C, corresponding to 740 mm.Hg vacuum This is hard to overcome unless a more effective vacuum pump is available. However this is not much necessary in economic consideration. Moreover the use of solvent or light flushing oil in cleaning sludge in the vacuum pump might cause higher vapor pressure which then lowered the high vacuum.

7. The vacuum in the evaporating chamber decreased from the beginning and then was constant after a period of operation. This could be said that at first the plate did not gain heat enough to evaporate water successfully, the evaporation then took place at a low rate. After the plate gained more heat (by being contacted

with circulated hot water), the evaporation took place at a higher rate. The vapor pressure in the evaporating chamber increased, the vacuum decreased. This was supported by the observation of low temperature at the entrance of vapors into the condenser and its increasing gradually. Furthermore, some water vapor from the evaporating chamber might not be condensed in both condensers. It then passed through the vacuum pump and result in lower vacuum.

8. The viscosities of the evaporated orange juice was seen to change point to point along the plate. The more viscous juice developed from the entrance to the end. The juice load at the beginning was greater than that at the end since water was removed during evaporation. This also made uncontinuity flow of juice. The assumption that the viscosity, and the density were constant might be disappointed. According to the physical properties determination of orange juice at various concentration, the orange juice changed from Newtonian fluid into Non-newtonian fluid at concentration about 20° Brix. (see Figure 13). The plate, to serve this correspondance, might have its inclination in exponential form, so the flow at the beginning may be slowed, and when the fluid was more viscous the flow must be accerelated by gravitational force.

9. The cleaning of the evaporating chamber was difficult since the chamber was closed. Some particles were left and dried on the plate which interfered the continuity of flow of the liquid film.

10. Too high pressure of steam (more than 4 lb/in^2) used in the heat exchanger seemed to be worthless, because the hot water

expanded too much and left the hot water cycle via a vent. Moreover, the high temperature of hot water resulted higher temperature of the plate than required to evaporate water, less effectiveness of the plate occurred because of the existance of dried spots on the plate.

5.6 Physical Properties Determination

The physical properties of orange juice : density, viscosity, and heat capacity at various concentrations were determined. The experiments were all taken at room temperature.

5.6.1 Determination of orange juice density (ρ) at various concentrations

By weighing a definite volume, 25 ml. using a graduated pipette, of various concentrations of orange juice, 5, 10, 15, 20, 25 30, 35, 40, 45, 50° Brix, the densities were calculated as gm/ml. The relationship between the density and concentration (Brix) was plotted as shown in Figure 12.

5.6.2 Determination of viscosity (μ)

Orange juice of concentrations 5, 10, 15, 20, 25, 30, 35 40, 45, and 50° Brix were prepared. The viscosities were determined by using Brookfield viscometer model LVF-5X with spindle NO.1 at speed 30 and 60. The figure read were multiplied by factors 2 and 1 respectively as indicated by the instruction of the viscometer. The results expressed as centipoise were plotted against concentration as shown in Figuro 13.

5.6.3 Dotcrmination of heat capacity (s)

The heat capacity at each interval of concentration was determined by mixing a known amount of hot water with a known amount of orange juice in a closed thermos, whose heat capacity was determined before. From the temperatures of orange juice, of hot water before mixing, and that of after mixing, the heat capacity was then calculated. Tho results of heat capacity (Cal/g^{o}C) were plotted against concentration as shown in Figure 14.

5.6.4 Brix and concentration

This expression is used rather loosely throughout the fruit juice industry. Primarily it is a measure of sugar (sucrose) concentration as determined by a Brix hydrometer which is calibrated to read directly in terms of percent weight by weight of sugar, and as such should only be used for pure sugar solution.[1] The experiment was carried out to ensure that the concentration of pure sugar solution would be represented by the reading of hand refractometer and these results were shown in Figure 15. Further assumption was made that the brix reading showed percent weight by weight of total soluble solids (or solid content) in orange juice.

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[1] Anthony Woollen, Food Industries Manual (20thed. London: Leonard Hill, 1969), p.245.

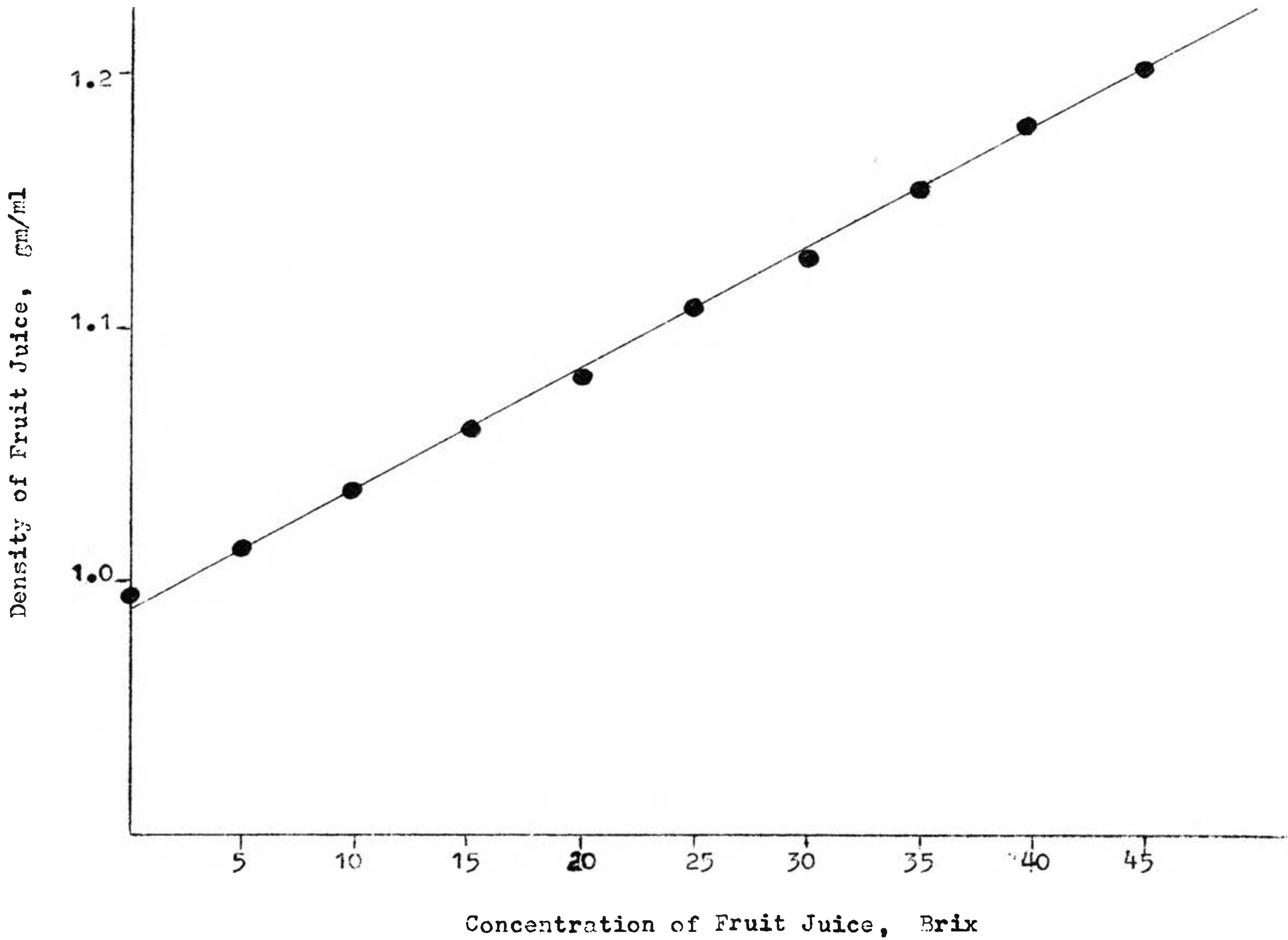

Figure 12. Variation of Density of Fruit Juice with Concentration

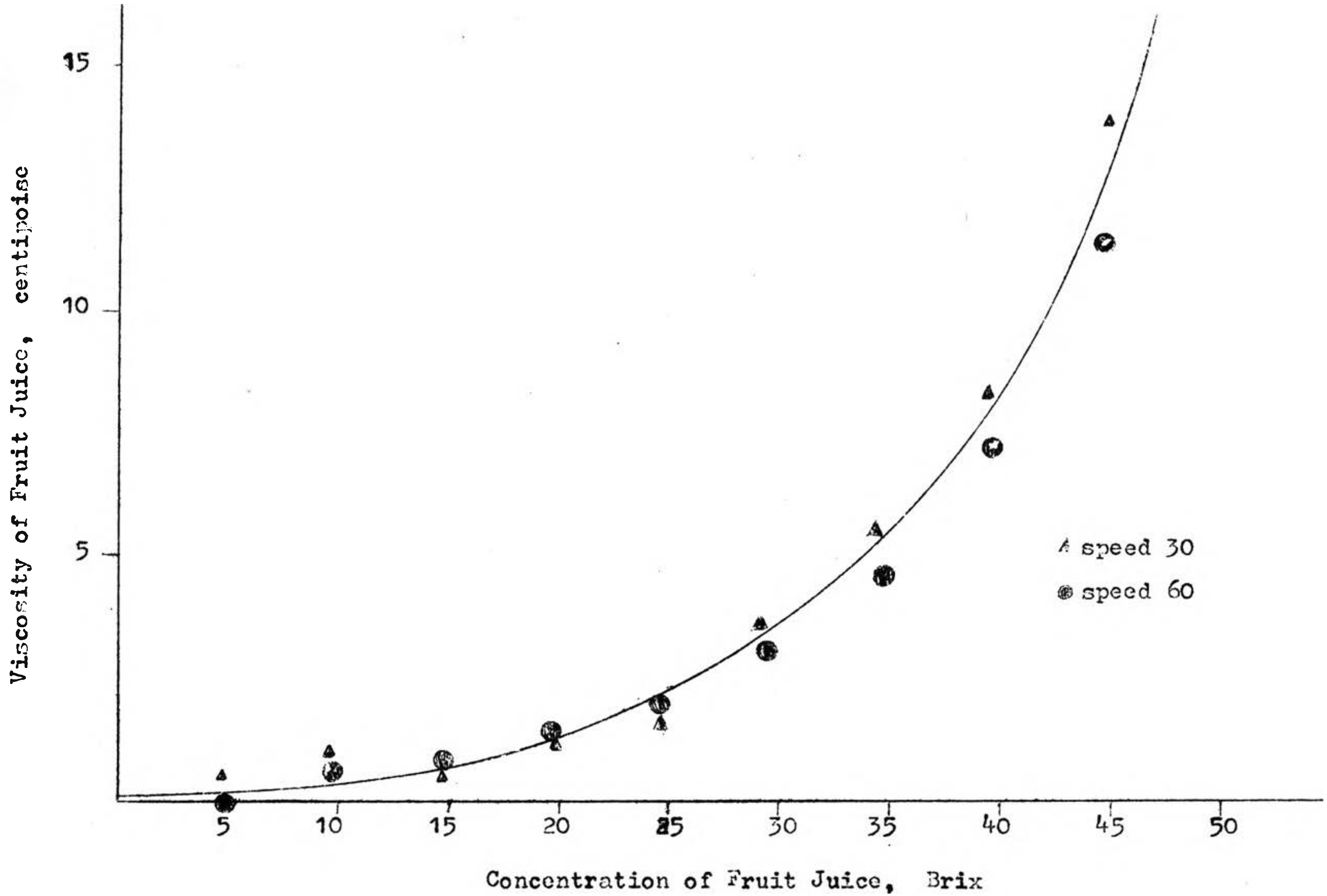

Figure 13. Variation of Viscosity of Fruit Juice with Concentration

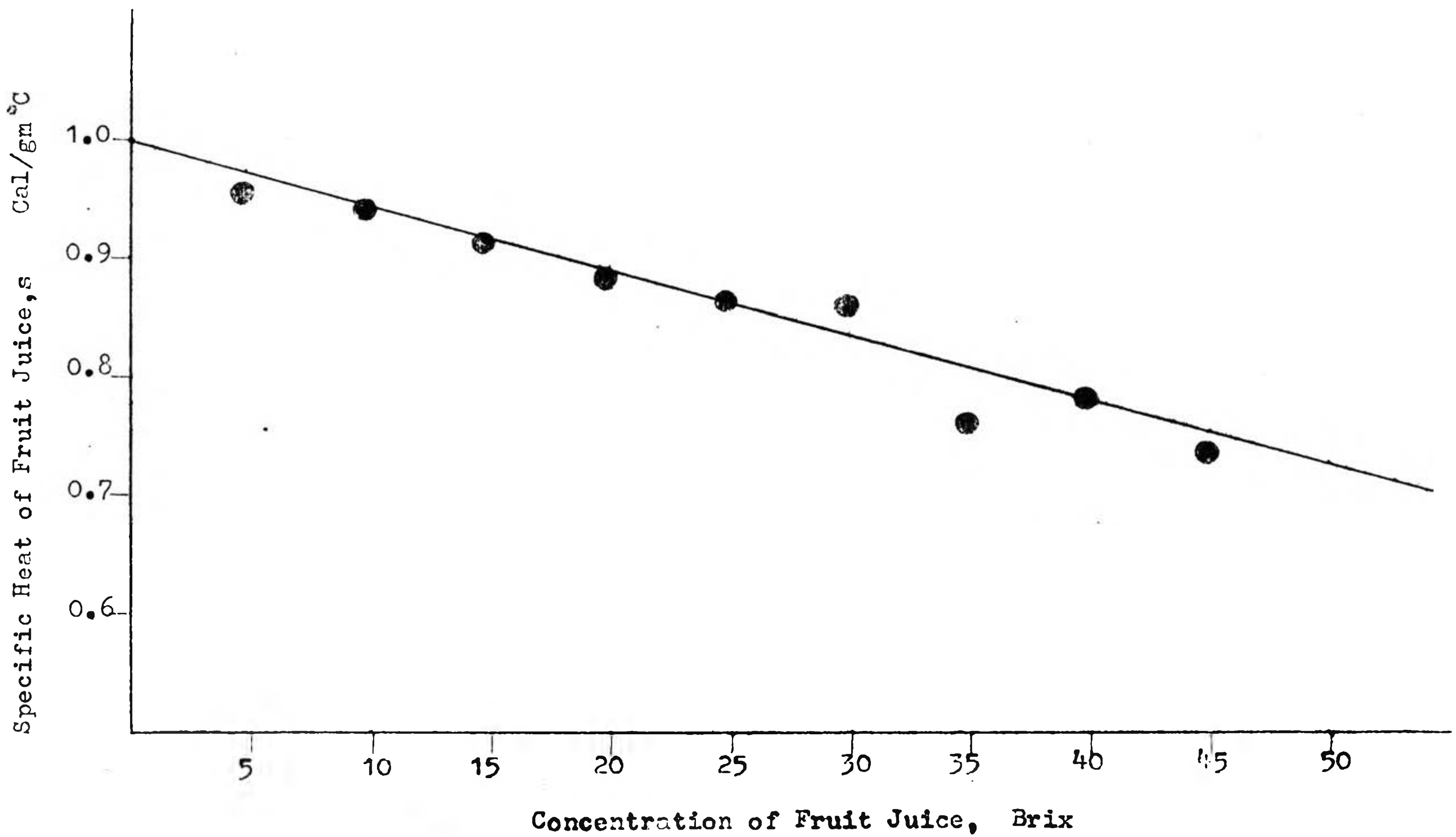

Figure 14. Variation of Specific Heat of Fruit Juice with Concentration

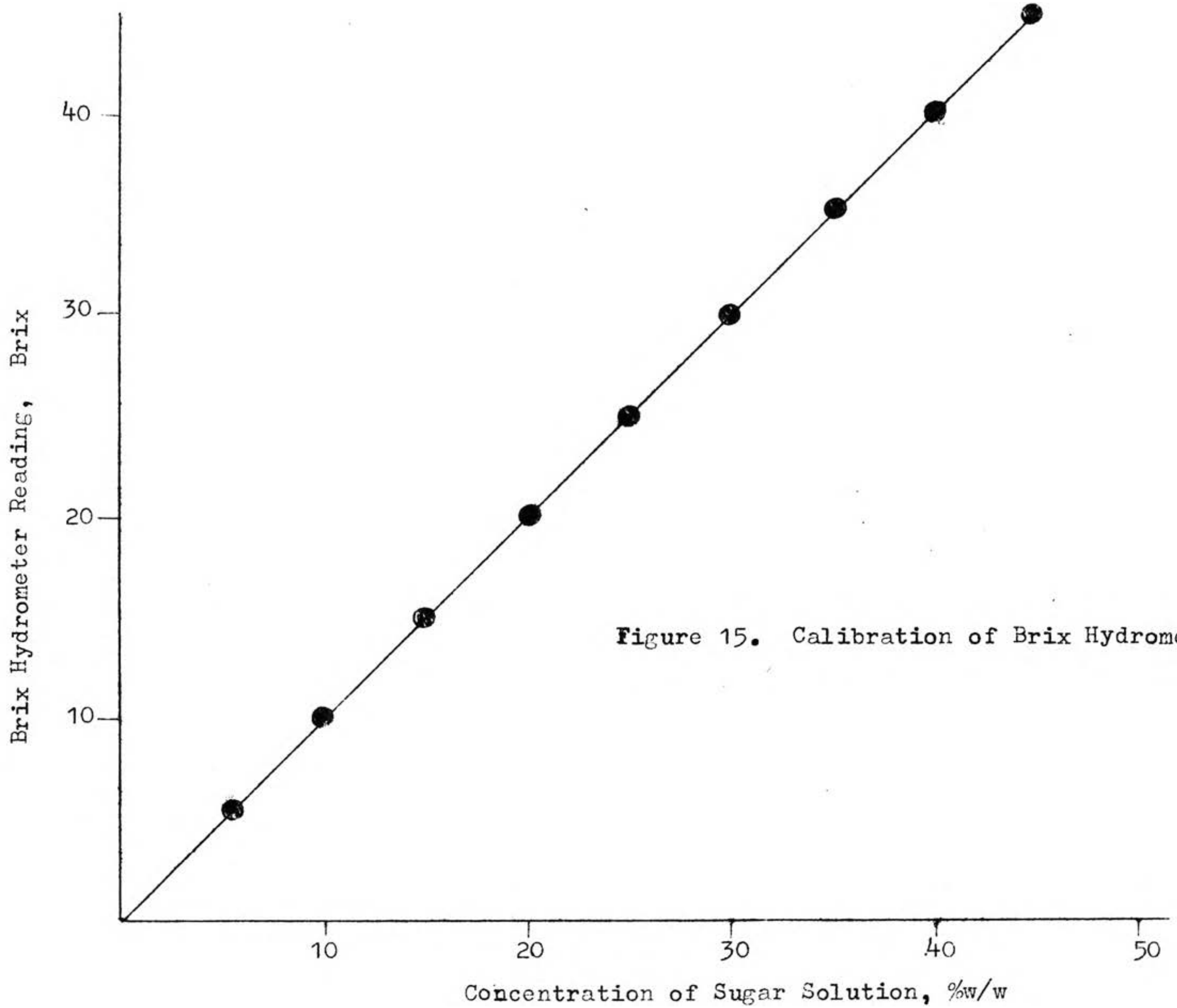

Figure 15. Calibration of Brix Hydrometer