TECHNICAL REPORT
71-7-FL

AN ATMOSPHERIC STEAM SUPERHEATER FOR STEAM DISTILLATION OF FOOD ANTIOXIDANTS

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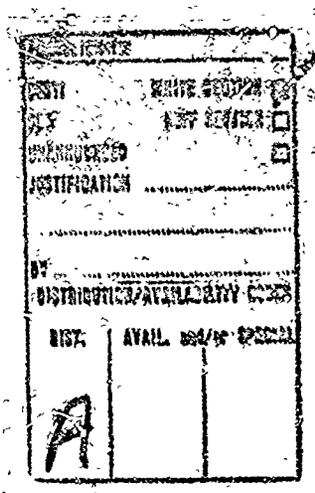
UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760

Food Laboratory
FL-109



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FOREWORD

The quantitative analysis of fats and oils for their antioxidant content has been described by Anglin, Mahon, and Chapman (1) and Eastman Food Laboratory(2). Both procedures require steam distillation, but Anglin, et al(1) shows a preference for superheated steam to maintain a temperature of 160°C. without the precipitation of salts in the sample.

It was decided after some investigation by the Analytical Group, Food Chemistry Division to use the method of Anglin, et al in the analysis of the antioxidants BHA (butylated hydroxyanisole), BHT (butylated hydroxytoluene) at Natick. The foregoing group requested the design of a steam superheater with a capacity large enough to supply superheated steam for the simultaneous analysis of twelve samples.

It was also agreed that, in the interest of laboratory safety, the steam superheater would operate at atmospheric pressure.

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ABSTRACT

A steam superheater was designed and built to operate at atmospheric pressure and to provide superheated steam for as many as twelve edible fat and/or oil samples undergoing steam distillation for BHA and BHT antioxidants. The steam flow rate is sufficient to yield a distillate flow rate of 4 ml/min for each sample. Superheated steam is injected at a high enough temperature to maintain the sample mixture at 160°C. with little or no change in volume.

INTRODUCTION

The report of Anglin, et al(1) describes a steam distillation technique for analyzing the antioxidant content of edible fats and oils. Essentially, superheated steam is passed through the sample solution containing an antioxidant such as Tenox BHA*, or BHT**, and the antioxidant is carried over with the distillate which is then analyzed spectrophotometrically for antioxidant content.

The high temperature steam required by the above procedure could be supplied by a commercially available, miniature steam generator. To obtain the desired temperature in this manner, however, would involve objectionably high pressures. The alternative to a pressurized steam generator is an atmospheric superheater. From the standpoint of safety and maintenance, the atmospheric superheater has clear advantages.

After reviewing the commercial availability of atmospheric steam superheaters, it was decided that a steam superheater could be built to do a better job at a lower cost than anything available on the market.

* butylated hydroxyanisole
** butylated hydroxytoluene

Tenox is a registered trademark Eastman Chemical Products, Inc.

DESIGN AND FABRICATION

The major elements of the superheater are the packed shell, the strip heaters, and the controllers (Figure 1). The shell consists of a thin-walled stainless steel cylinder 7.5cm in diameter and 90cm in height. There is nothing critical about these dimensions - this section of tubing just happened to be available. The ends are sealed with a welded in-place blind member on the bottom and a bolted bonnet on top. Twelve 1/4" OD welded stainless steel nipples are equally spaced around the circumference near the top to provide outlets for the superheated vapor. Other welded nipples provide connections for feed water, a drain line and thermometers. The shell is packed with 5/8: thin-walled stainless steel rings.

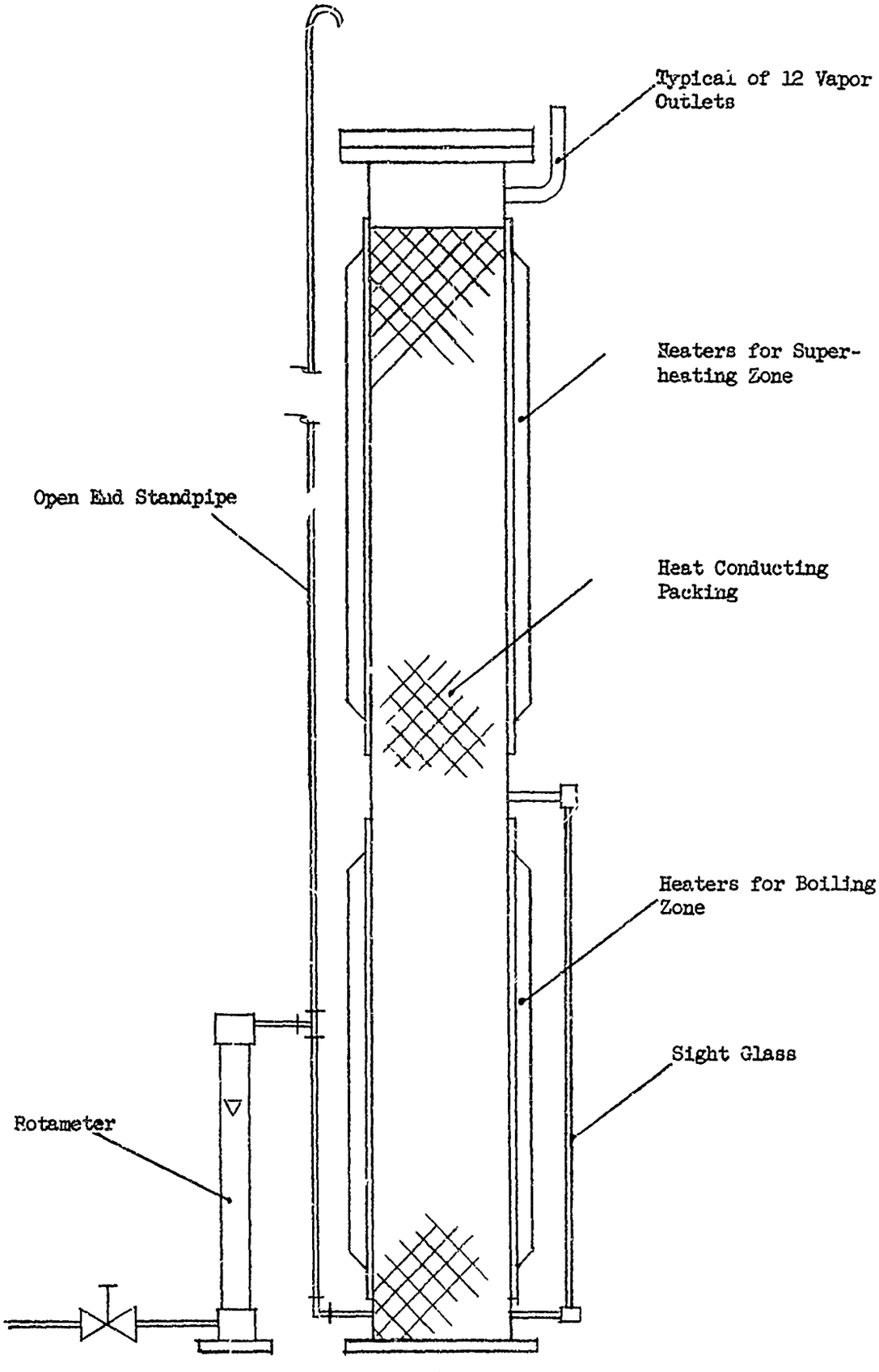
Two sets of ordinary strip heaters are strapped longitudinally to the outside of the shell - one set on the lower section and the other above. There are 4 heaters in each set and all are bedded in putty* to expedite the transfer of heat. The 4 units in each set are connected in parallel and supplied with 208-volt single phase power through separate controllers. Boiling and superheating can be regulated individually with this arrangement. The combined heating capacity of the four SE 1801 heaters** in the lower set and the SE 1205 units** in the upper set is respectively equivalent to 3,000 and 1,500 watts at the indicated voltage.

The column and heater assembly is housed in a rectangular enclosure made of one-inch block magnesia.

De-ionized feed water is introduced through a rotameter*** at a rate to correspond with the superheated steam demand. It enters the lower part of the column at room temperature and is almost immediately flashed to vapor. The vapor is then superheated to 265°C. as it rises through the column at a pressure in the order of ounces/square inch. It is then delivered through insulated Teflon tubes of equal length to each of the analytical assemblies. The hot vapor enters the analytical apparatus through an 1mm orifice at a high enough temperature to permit steam distillation at 160°C.

* Thermon - T-63
** Chromalox Cat. No.
*** Matheson (No. 702-B5V)

FIGURE I
SCHEMATIC ARRANGEMENT OF SUPERHEATER



There is a 6-foot open-ended standpipe between the rotameter and the column. Its purpose is to provide a static hydraulic head to overcome the pressure drop in the system and to provide an unrestricted vent for safety. Each set of heaters is equipped with a separate temperature controller. The controller for the lower heaters is an inexpensive bimetallic timer type* which is simply used to control the boiling rate and, consequently, the level of water in the lower part of the column. The level, however, is relatively unimportant as long as it is somewhere near the center of the heater span.

The controller for the upper set of heaters is a sensitive thermistor type**, the probe of which is located in the headspace near the top of the column. This unit is capable of maintaining the temperature of the superheated vapor within 3°C. of a set point after the system has become stabilized.

Miniature indicating dial type thermometers*** are used to observe the temperature in both the boiling and superheating zones.

Appendix B includes a photograph of the superheater and the satellite distillation apparatus.

EXPERIMENTAL PROCEDURE

The procedure used in the quantitative analysis of BHA and BHT is that of reference(2) with the exception that superheated steam is used instead of saturated steam at atmospheric pressure. The superheated steam is injected into the antioxidant sample solution at a high enough temperature to support steam distillation at 160°C. The antioxidants are readily distilled from the oil or fat samples under these conditions. The advantage of using superheated steam as opposed to saturated steam is that the optimum temperature for distillation can be achieved at a faster rate and that the temperature and volume are subject to better control.

Experimental runs were made using the same procedure, distilling eight samples at a time. The superheater is designed to accommodate 12 analytical systems but the apparatus for only eight is presently available. The distillate flow rate and temperature of each sample was measured and recorded for later data analysis. At the end of the runs, the distillates were analyzed for BHA and/or BHT. In addition,

* Chromalox Cat. No. Ch-252
** Chromalox Cat. No. 53T-1
*** Weston Cat. No. 2291

four runs were made on the same sample to check experimental reproducibility.

Appendix A includes the operating procedure for the superheater.

DATA AND ERROR ANALYSIS

1. Steam Distillate Flow Rates:

The distillate from each sample undergoing antioxidant analysis was collected during the 30-minute distillation period. The volumes collected from a typical 8-sample run were:

SAMPLE	VOLUME
1	122.9
2	120.6
3	120.4
4	121.2
5	121.0
6	122.2
7	122.4
8	121.6

These figures, as percent deviation from the average, are graphically presented in Fig. II.

2. Heat Balance:

The steam leaves the superheater at 265°C. but the temperature is probably considerably less as it enters the sample mixture because of heat losses along the way. The actual temperature of the steam as it enters is irrelevant since it has been established that a heat balance is obtained with 265°C. steam at the superheater and a 100-watt input to the mantel of the distilling flask. Under these conditions the volume of the sample mixture remains constant and the temperature can be maintained within ± 2° of 160°C. Fig. III shows the temperature change of a typical sample mixture with respect to time.

FIGURE II

DEVIATION FROM AVERAGE DISTILLATION
RATE DURING 30-MINUTE PERIOD FOR
TYPICAL SAMPLE ARRAY

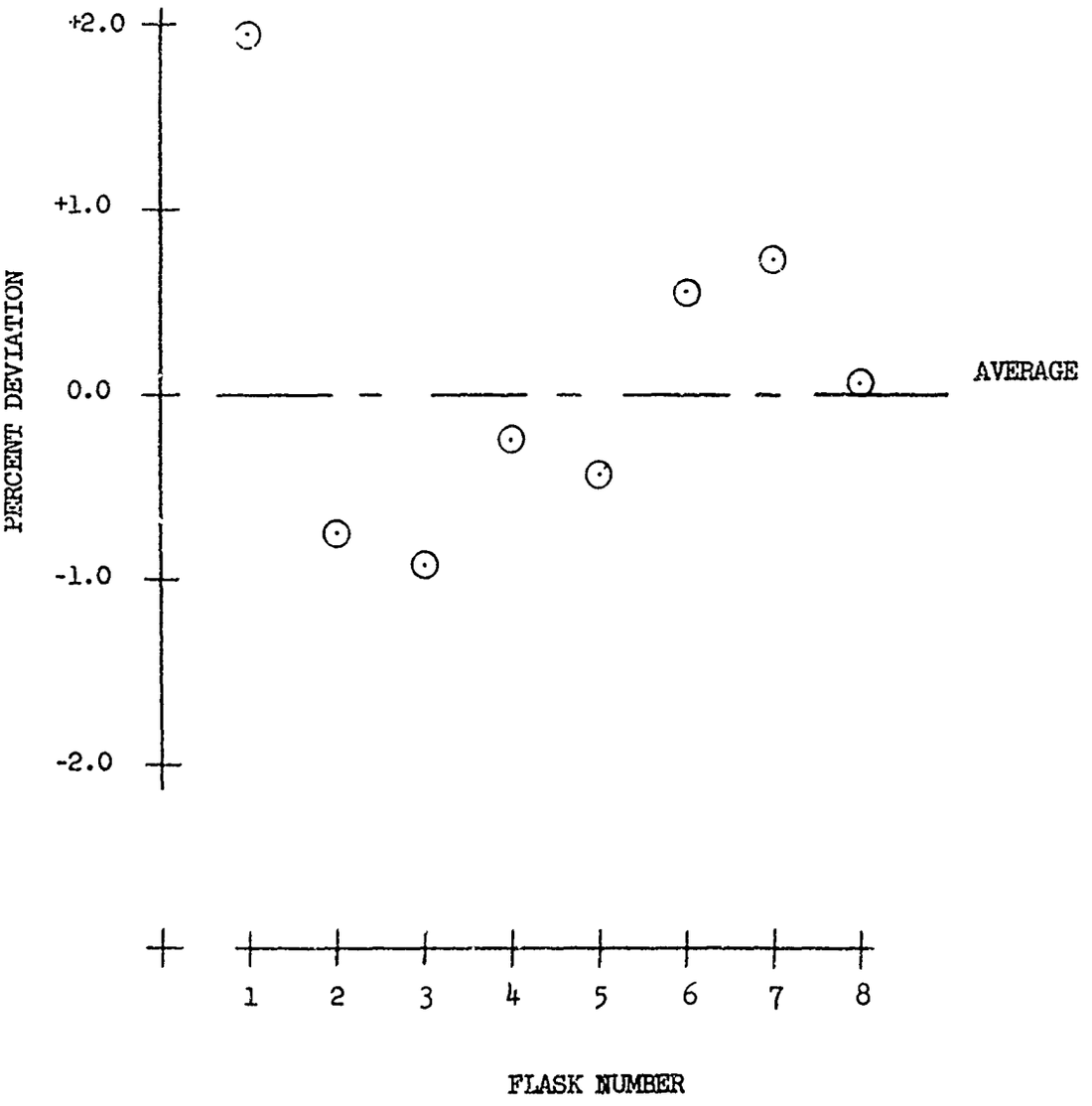
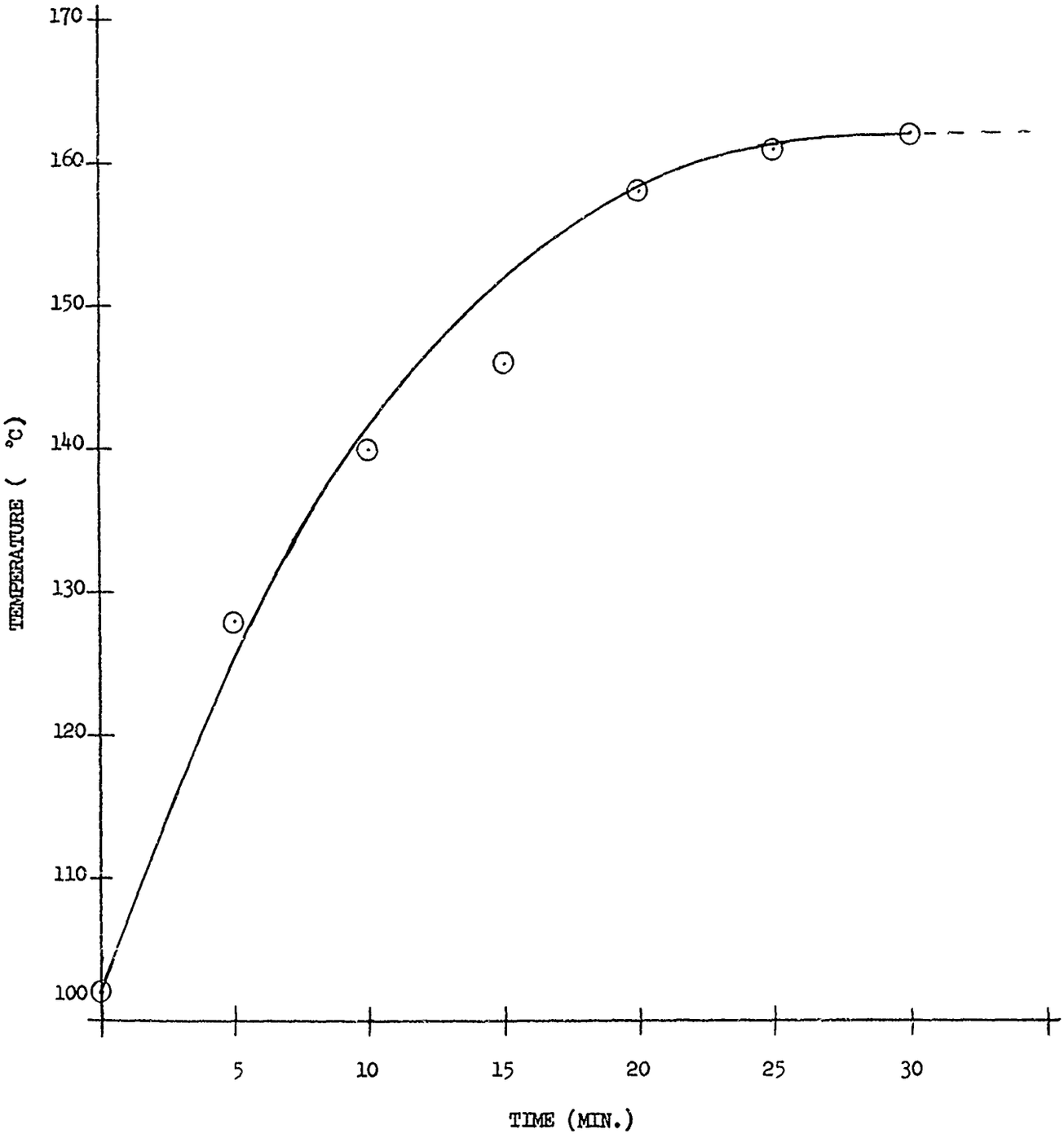


FIGURE III

TEMPERATURE OF SAMPLE
MIXTURE VERSUS TIME

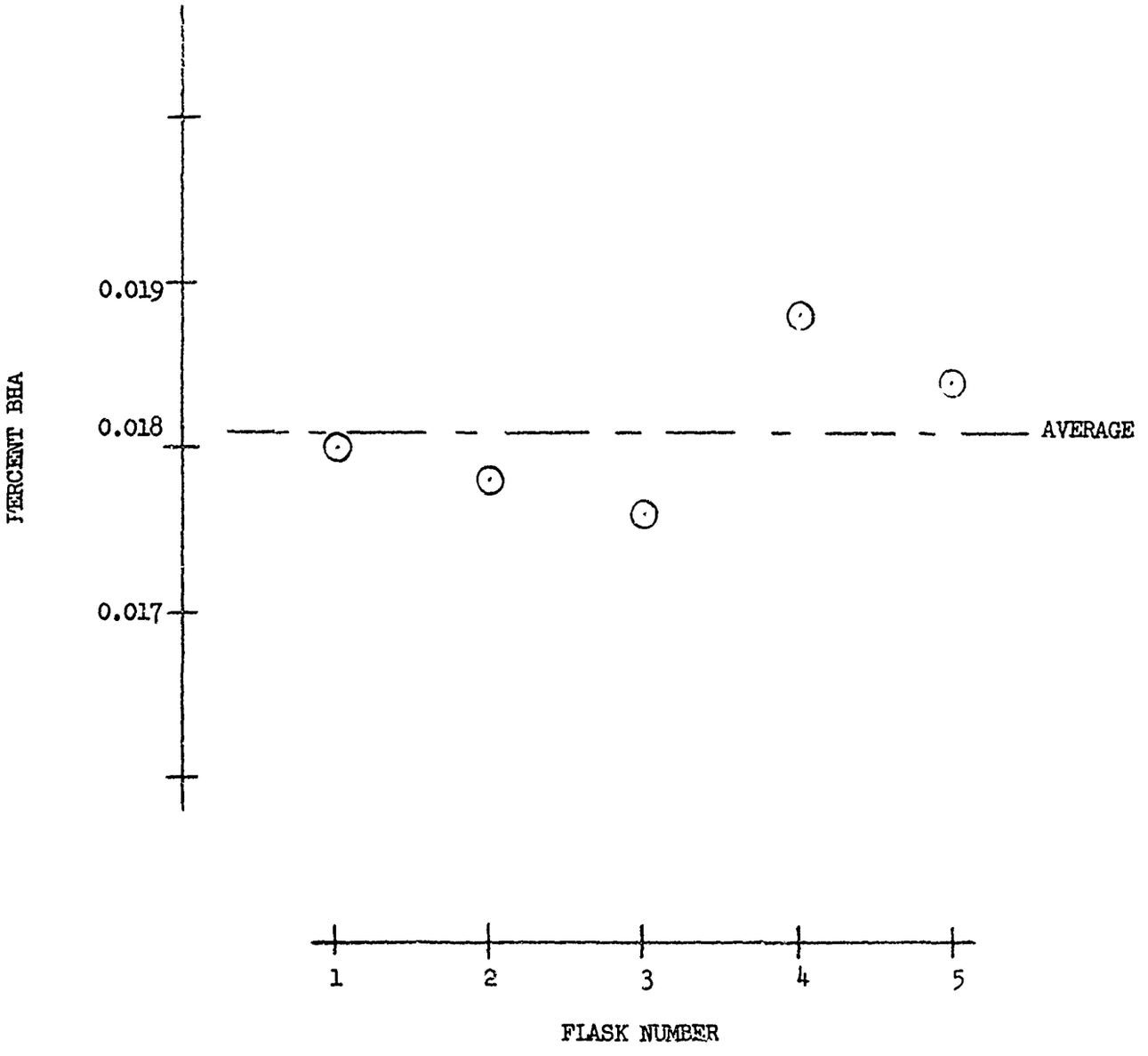


3. Reproducibility of Antioxidant Determination:

The real test of any analytical technique is the reproducibility. To test the method of steam distilling BHA and BHT antioxidants, four runs were made on aliquots of the same fat sample. The reproducibility for a fat sample containing approximately 0.019% BHA is ± .001 percent (Fig. IV). This is an error of ± 5% which is acceptable for antioxidant analysis.

FIGURE IV

REPRODUCIBILITY - STEAM DISTILLATION
OF FAT CONTAINING BHA (ALIQUOTS TESTED SIMULTANEOUSLY



SUMMARY

The steam superheater designed and built to provide superheated steam for the steam distillation of antioxidants performs satisfactorily with good reproducibility.

It requires 20 minutes from a cold start to generate superheated steam at a temperature of 265°C. At this temperature and with a 100-watt input to the mantle of the distillation flask the sample mixture can be maintained within ± 2° of 160°C. with little or no change in volume during the 30-minute distillation period.

The superheater is designed to serve as many as 12 distillation systems simultaneously. Results with eight distillation systems indicate that the distribution of the superheated vapor between individual systems is satisfactory.

LITERATURE CITED

(1) "Determination of Antioxidants in Edible Fats", Anglin, C., Mahon, J.H., and Chapman, R.A., J. Agr. Food Chem. 4, 1018(1956).

(2) "Quantitative Analysis of Fats and Oils for BHA and/or BHT by Steam Distillation", Eastman Food Laboratory Standard Procedures No. 18 and 18A, Aug. 15, 1967/TDS G-162.

APPENDIX A

OPERATING PROCEDURES

FOR

LABORATORY STEAM SUPER HEATER

Start-up

1. Fill the lower or boiling section of the super heater to the mark on the sight glass (or within one and one-half inches from the top). When this level is reached, shut the rotameter off and initiate the boiling by turning the lower strip heater control switch to "HI".

2. When the level in the sight glass begins to surge, open the rotameter to the desired setting (see Table I), and adjust the lower strip heater control to maintain a constant level in the sight glass.

3. After 15 minutes of operation, turn on the upper strip heaters and set the thermostatic control to 265°C.

4. An additional 15 minutes is required for temperature in the upper half of the super heater to stabilize. At the end of this time, a final temperature check should be made on the dial thermometer located at the top of the super heater. If the chamber temperature is 265° ± 5°C, distillation may be started.

EMERGENCY PROCEDURES

In the event of a loss of water flow to the rotameter, both the upper and lower strip heaters should be shut off.

SHUT - DOWN

1. Shut off both the upper and lower strip heaters.

2. Close the rotameter valve and shut off the valve controlling the water flow to the rotameter.

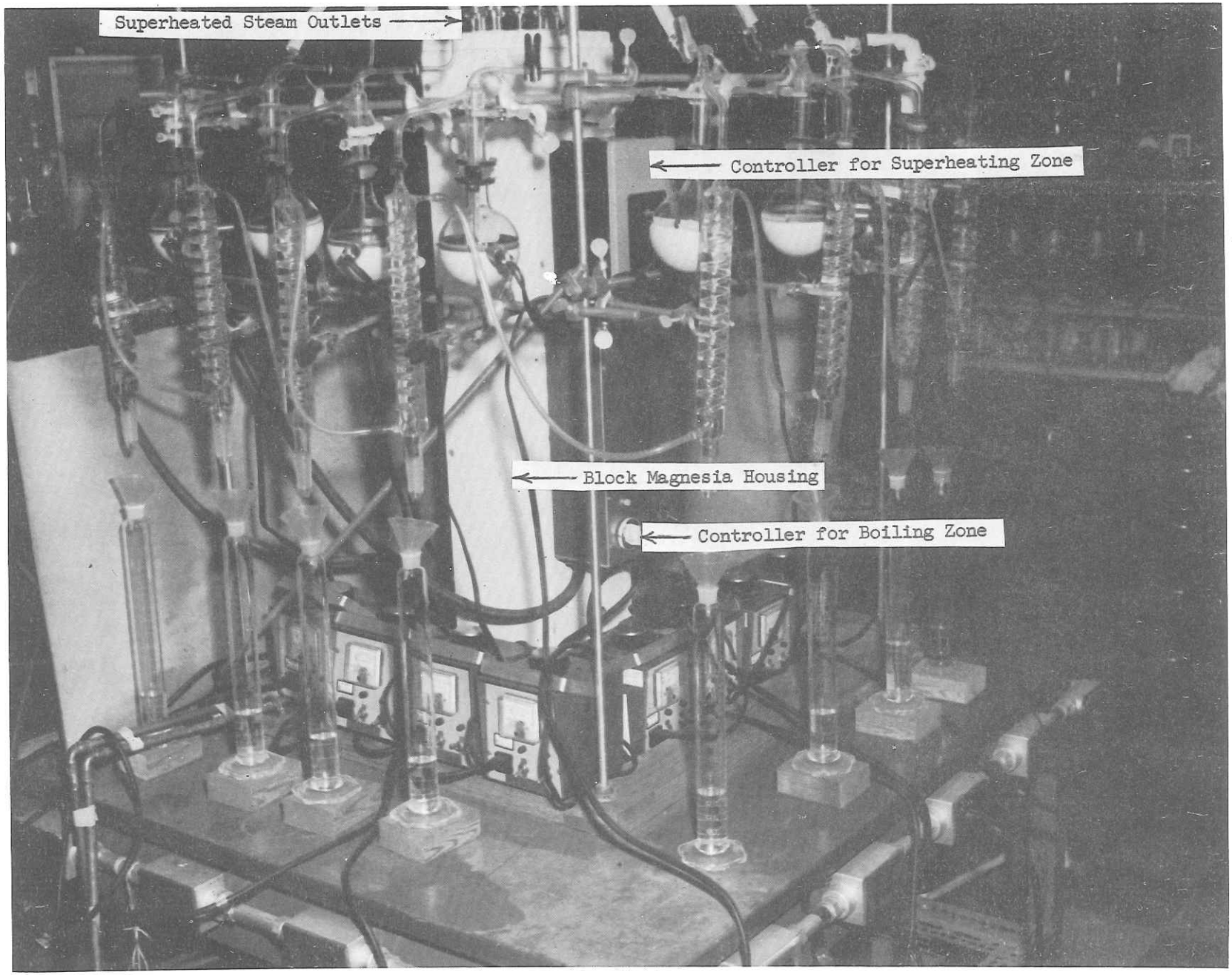
TABLE I

Number of Distillation Units	Rotameter Setting
4	6.5
5	7.5
6	9.2
7	10.2
8	12.2
9	13.0
10	16.5
11	19.0
12	21.5

NOTE:

Based on four ml. water vaporized per minute per distillation unit.

APPENDIX F



ATMOSPHERIC STEAM SUPERHEATER AND BHA DISTILLATION APPARATUS

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	ROLE	WT	ROLE	WT	ROLE	WT
Design	8					
Superheaters	9					
Analysis	4					
Oils	4					
Fats	4					
Antioxidants	4					