Improved Design of the Active Oxygen Stability Apparatus

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THE A. O. M. stability apparatus while having certain inherent faults, both in principle and in operation, has come to be accepted as the most conventional and dependable method of measuring the stability of animal and vegetable fats and oils. The unit as originally designed and reported (1) has undergone little mechanical change (2, 3, 4).

The method consists of bubbling air into hot fat (208°F.) until a certain concentration of organic peroxides are formed in the fat as determined by titration. Essentially all that is required is a means of holding the fat at the desired temperature and the introduction of clean air at a reasonably constant rate into the fat.

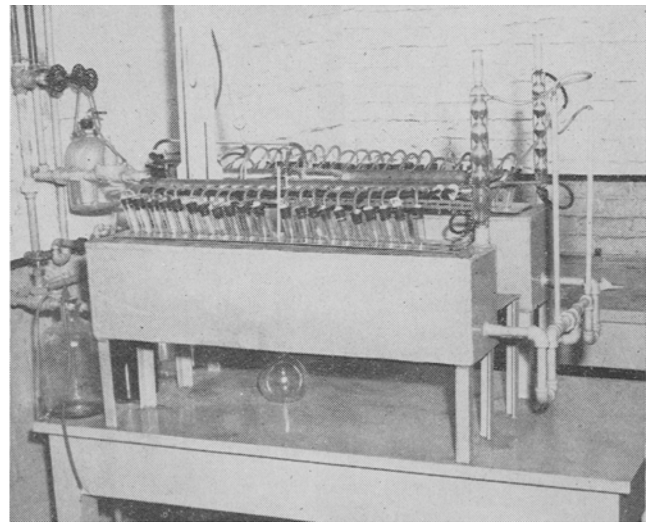
The commercial design is either gas or electrically heated, and with the use of an oil bath potential fire hazards exist unless an attendant is always present. This feature practically eliminates overnight operation and with a shortening of two to three hundred hours stability, an extended period is required (if operated only 8 hours per day) to obtain results.

The air distributing system is a complex assortment of tubing and bottles with one tube running eventually to each sample of fat under test. Pressure variations cause broken and disconnected tubing, resulting in floods and interrupted operation. The scrubbing solutions tend to evaporate.

Tube cleaning has always been a problem, although some improved designs have been reported (3). Mehlenbacher (5) has reported on methods of accelerating the test by operating the unit at a higher temperature (110°F.), but inasmuch as the reproducibility of results at the lower temperature is at about a 10% level, the higher temperature should tend to give even greater experimental errors.

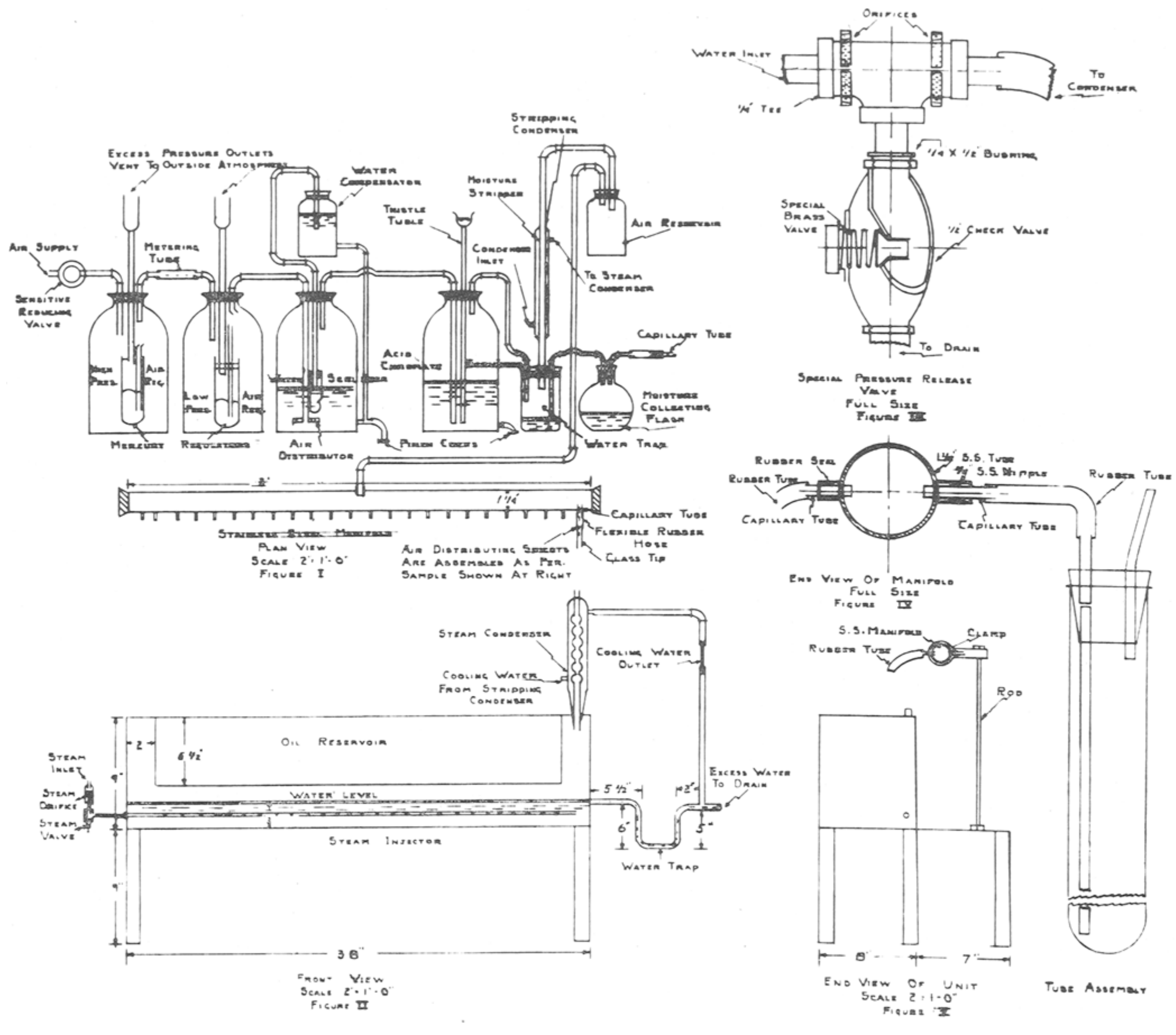
We have designed and constructed a unit in which most of the undesirable features have been eliminated. The unit may be constructed very economically. A twin unit (24 tubes each) has been in constant operation in our laboratory for 6 months and has given very satisfactory service and results. The unit is steam heated.

The bath, Figure 2, was constructed of 10-gauge steel plate, and the steam is introduced at one end through a calibrated orifice before entering a perforated pipe in the bottom of the bath. On condensing, a water level of 3 inches is maintained. A means of draining has been provided. The oil reservoir is approximately 2 inches above the water level, and the conventional type tube holder and oil bath is used. A low resistance bulb type condenser is used to reflux the steam. This heating system, with steam entering the bath at atmospheric pressure, maintains a uniform temperature of 208°F. indefinitely. A recording thermometer may be inserted in the bath and a temperature recording made when the unit is unattended. We have noted no factors to date which would affect the temperature except actual clogging of a steam line somewhere external to the apparatus.



While it is well known that the rate of air flow through the fat is not critical (6), a reasonably constant rate is desired. Constant reduction of the house pressure is necessary to eliminate tube "blow off," etc. In our unit (Figure 1) air is introduced by a sensitive reducing valve into a high pressure regulating bottle. The excess air passes through the mercury in the small tube to the outside atmosphere. The air at reduced pressure passes through a metering tube into the low pressure bottle, where the excess is again vented to the outside atmosphere. It then passes into the water scrubber through a perforated tubing which gives greater diffusion. The level of this bottle is kept constant by the water compensator bottle which is shown. The air then passes through the conventional acid chromate scrubber. A thistle tube has been attached for conveniently adding solution. After scrubbing, the air passes through a trap and stripping condenser to the air reservoir and some excess moisture is removed. While the effect of moist air on the results in general is debatable (7), the condensation of water in the manifold is not desirable.

A stainless steel manifold (Figure 1, also shown full size in end view), was constructed of 1¼ I. D. pipe stoppered at both ends with rubber stoppers. One-fourth inch stainless steel nipples (inside dia.) are threaded into the manifold at intervals. The capillary tube is fitted into a short length of rubber tubing and the assembly placed in the nipple. A short length of rubber tubing is then attached to the capillary tube (about four inches) and a short two-inch inlet tube to the test tube holding the fat. This glass inlet tube only penetrates one-half the length of the rubber stopper of the fat test tube, permitting easy and complete removal of all tubing for cleaning. The air inlet tube also only penetrates one-half of the length of the stopper and can easily be removed for cleaning. The effluent tube is un-



IMPROVED STABILITY APPARATUS

TABLE I

LOW STABILITY LARD

	Peroxide titrations at		
	100 min.	200 min.	240 min.
	me	me	me
Normal rate	14	26	45
50% above normal	15	30	50
50% below normal	14	30	47

MODERATE STABILITY LARD

	Peroxide titrations at				
	60 min.	120 min.	180 min.	200 min.	240 min.
	me	me	me	me	me
Normal rate	14.5	16.0	16.5	16.5	18.0
50% above normal	15.0	16.5	16.5	17.5	22.0
50% below normal	15.0	17.0	19.0	20.0	23.0

MODERATE STABILITY HYDROGENATED FAT

	Peroxide titrations at			
	0 hrs.	20 hrs.	22.5 hrs.	23 hrs.
	me	me	me	me
Normal rate	13.5	39	49	51
50% above normal	13.5	42	54	60
50% below normal	13.5	48	60	70

changed. The capillary tubes in each nipple may be calibrated in the usual manner.

Some results of varying the air rate are hereby listed (Table I). It can readily be seen that air rates 50% above the prescribed one had no significant effect on the results, however, using flows 50% below normal gave slightly higher peroxide titrations in some cases. This is undoubtedly due to a slower removal of the more volatile organic peroxides by the reduced flow.

In Figure 3 is shown a specially designed valve fitted into the line through which the water for the two condensers passes. This valve permits great fluctuations in water pressure in the house system without blowing the rubber tubing off the inlets to the condensers, as all excess water passes to the drain.

The air distributing manifold may be mounted as shown in Figure 4 and the air regulating bottles, etc., on the platform behind the bath or on a table.

Summarizing, we have made certain mechanical improvements in the Active Oxygen Apparatus which permit uninterrupted, unattended over-night operation. These may be listed as follows:

1. An apparatus designed for and heated with steam.
2. An improved air regulating system.
3. An improved air distributing system using a stainless steel manifold.
4. A safety valve is shown which eliminates the possibility of leaks in the water condensing system.
5. A different type of aeration tube assembly is described.

We are indebted to Mr. H. Flagler of the Research Engineering Department for the sketches inserted.

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The Effect of Sodium Carboxymethyl Cellulose on Synthetic Detergent Systems

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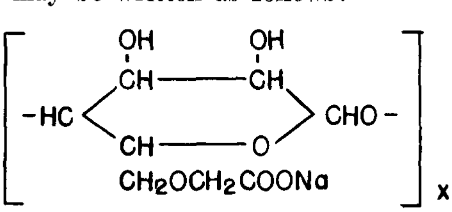
Introduction

FOLLOWING the termination of World War II in the European Theater, Hoyt, investigating certain phases of German industry, reported that certain forms of sodium carboxymethyl cellulose had been used in large quantities as a soap and synthetic detergent extender (1). Detailed information concerning its manufacture and properties and samples of one product, Tylose HBR, were sent to this country. At that time these laboratories held a contract with the Office of the Quartermaster General covering research on the development of laundering procedures and detergents suitable for use with sea water. In this connection Tylose HBR was evaluated for detergency promoting properties on three commercial synthetic detergents: "Arctic Syntex L," "Igepon T," and "Neutronyx 228," as hereinafter reported. Upon completing this sea water laundering investigation, attention was directed to the formulation and detergency evaluation of compositions based on a sodium alkyl aryl sulfonate type detergent and sodium carboxymethyl cellulose.

Although in this country sodium carboxymethyl cellulose is used in ice cream manufacture, textile finishing, paper sizing, and other equally diverse industries, its applicability to the field of detergency, judging from published information, appears relatively unknown. It is the purpose of this paper to present some new data on the effect of sodium carboxymethyl cellulose on the carbon soil removal and whiteness retention characteristics of several built and unbuilt synthetic surface active agents and to discuss effects of the material such as water softening properties which are of importance in the field of detergency.

Chemical Structure and General Properties of Sodium Carboxymethyl Cellulose

The chemical structure of sodium carboxymethyl cellulose may be written as follows:



In this idealized representation the glucose residue which forms the basic unit of the cellulose molecule is shown to be substituted in the primary hydroxyl group. The two secondary hydroxyl groups may similarly be substituted and the extent to which the cellulose is modified is expressed in terms of degree of substitution. If on the average one hydroxyl group in each glucose residue unit is substituted, this is termed one degree of substitution.

Sodium carboxymethyl cellulose in purified form is a white odorless and tasteless solid. It is believed to be physiologically inert (2). Its solubility in water varies with the degree of substitution; in general, the greater the degree of substitution, the greater the solubility. An extensive discussion of the chemistry of this material is given by Höppler (3), and a review covering properties, manufacture and uses, has been contributed by Hollabaugh *et al.* (4).

The Measurement of Detergency

In evaluating the effect of sodium carboxymethyl cellulose on synthetic detergent systems, our work largely has been confined to the experimental laundering of cotton fabrics. For satisfactory laundering such fabrics require highly effective detergents and therefore make excellent test material. It is our belief that detergency with reference to cottons may be considered in two phases. One—soil removal—applies to the capacity of a detergent solution to remove soil from a standardly soiled fabric; the other—whiteness retention—involves the capacity of the solution to suspend a colloidal soil and prevent its deposition upon an unsoiled standard fabric (5).

The laboratory methods used in determining the detergency data reported herein evaluate independently these two characteristics. They have not previously been published and accordingly are given in full in this section.

Pretreatment of Cotton Fabrics. Prior to use for detergency studies it is necessary to pretreat most cotton fabrics so as to remove the sizing or finish and to shrink the material.

In our work 14 pieces of a standard muslin[1] 36 in. x 54 in. are definished in a commercial monel metal wash wheel (24 in. x 34 in.) by the procedure indicated in Table I, using throughout water softened to below one

[1] Indian Head muslin (permanent finish) count 53 x 47, 5.15 oz. per sq. yd., manufactured by Nashua Mfg. Co., 40 Worth St., N. Y. 13, N. Y.