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Composting coffee waste

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COMPOSTING COFFEE WASTE

by

Lawrence James Hickey

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ABSTRACT

Disposal of waste, and specifically organic waste, has become a serious problem in such industries as the canning, meat-packing, dairy and other food producing industries. Recently, with the introduction of instant coffee powder, this particular industry now has a waste disposal problem with the resultant coffee residue. Composting or biologically decomposing this waste to yield an organic fertilizer is one of the many ways to solve this problem.

There are no papers in the literature concerning composting of coffee waste and only one paper (85) is available covering studies of high rate composting of garbage and refuse. It is known that work is being conducted by Wagner College and Michigan State College (46) in the decomposition of garbage on a pilot plant scale but as yet, no data has been published. Composting equipment has been patented by Earp-Thomas (13) (14), Taylor et al (77) and Eweson (21). However, none of these designs have proven to be practical on a commercial basis.

It is the purpose of this paper to study over a twelve hour period, the important composting variables associated with the aerobic, thermophilic decomposition of coffee waste. The aerobic process was selected because other composting investigators (13) (21) (46) (77) (85) advocated its use and because supposedly (31), the maximum decomposition of the organic matter is effected in the shortest possible time, process variables are easier to control and end products are odorless.

This study was successful in determining the optimum values (table XVIII) of the composting variables which are; air rate--3.6 cubic ft./lb.COD/hr., agitation--315 RPM., temperature--130°F., and the ratio of active to raw waste--2:1. Autoclaving of the raw waste almost triples the normal rate of decomposition. Under optimum conditions, disregarding waste autoclaving, the maximum reduction in oxygen demand was 7% in twelve hours and calculated to 8.5% in twenty-four hours. It is estimated that the bioxidation of coffee waste, if allowed to continue, would cease after 4 to 6 days with a maximum decomposition of 20 to 25%. This value is slightly lower than that of Wiley (85), who lists a 30% reduction in 6 to 9 days. The difference is due to; 1.) greater organic complexity of coffee waste, 2.) toxic materials such as formic acid and hydroquinone, 3.) the probable effect of coffee oil and fat coating micro-organisms and preventing them from functioning. Gurnham (27) reported that this latter condition existed in domestic garbage because of the fats and grease present.

Prior to undertaking this study, it was hoped that a 40% reduction of oxygen demand in twelve hours would be attained since it is necessary to have a final reduction of 50 to 60 percent in order to effect a satisfactory compost according to Martin & Waksman (45). Therefore, it is concluded that the aerobic process, which gives an estimated final reduction of 20 to 25 percent for coffee waste and 30 percent for garbage, does not produce an acceptable compost for commercial use.

It is believed that satisfactory composting of organic waste can be achieved by using the anaerobic bioxidation method. This belief is supported according to recent data assembled by Gurnham (27) and the present application of this method to produce compost from brewery (56) and meat-packing (41) wastes. It is recommended that the anaerobic method be investigated in the composting of coffee waste with regard to evaluating the variables of temperature, agitation, micro-organism concentration and waste conditioning.

This is the first paper to submit a method for solving the problem of coffee waste disposal. This is the first paper to study the variables of temperature, ratio of active to raw waste and waste conditioning by autoclaving in the aerobic bioxidation of coffee waste or any solid organic waste to produce compost. This is the first paper to use a modified form of the chemical oxygen demand method as developed by Pepinsky et al (55) to determine the rate of concentrated, organic waste decomposition which satisfies the demand for a suitable unit of measure.

INTRODUCTION

The process of composting can be defined as a method whereby organic waste such as leaves, manure, waste vegetables and industrial food waste are biologically decomposed to yield a fertilizer.

Until recently, the field of composting was confined to the practice of preparing compost piles on the farms from plant refuse or manure. At the present time, the field can

include, in the broadest sense, any process using aerobic or anaerobic bio-chemical oxidation to effect a product having fertilizing value.

Manure, a raw material for composting, usually refers to the solid and liquid excreta of livestock. For a long time, this livestock waste was the only source of organic fertilizer until it was discovered that any kind of plant refuse can be used to prepare an acceptable fertilizing product. This product prepared without the aid of animals can consist of straw, cornstalks, salt hay, garden rubbish, soy bean refuse, garbage that is free of metals and glass, grass clippings, leaves and needles from trees, weeds, peat, sawdust, various industrial by-products and many other kinds of plant waste can be used for making synthetic manure. Addition of certain chemicals containing nitrogen and phosphorus to the compost pile accelerated decomposition and improved the quality of the end product. However, even with these modifications, time of composting still consumed from 3 to 6 months of digesting.

The problem of disposal of industrial waste in the canning, meat-packing and coffee establishments demanded the development of an accelerated method for making compost. Compost piles did not satisfy this demand and in recent years the trend has been toward the adaptation of aerobic biochemical oxidation in the quest for a suitable method. In the last few years, a considerable amount of work has been done in evaluating the factors affecting aerobic bioxidation in the activated sludge process. Last year, it was revealed

by several colleges that they have developed similar aerobic methods which turn garbage and refuse into a plant food so rich that even mixed with 10 times as much ordinary sand, it is still better than average top soil. Although these methods are reported to be in the semi-commercial stage, there is no published data concerning their operational conditions. One investigator has studied the high rate composting of refuse and garbage and lists a 30% reduction of volatile solids over a six to nine day period. Various types of commercial composting equipment together with limited operating conditions have been designed but have not proven to be practical.

The development of the process of composting has been hindered by the lack of satisfactory design data concerning the variables involved. It is therefore the purpose of this paper to evaluate the important variables of temperature, air rate, agitation, micro-organism concentration and waste conditioning associated with the aerobic bio-chemical oxidation process. The organic waste to be composted is coffee residue made available by Maxwell House Division. Since embarking on the manufacture of powdered instant coffee, Maxwell House has been confronted with the problem of disposing of 30 million pounds of coffee waste per year.

All variables are investigated in a single piece of equipment called a digestor with regard to determining the optimum conditions necessary for composting the waste. Up to the present time, considerable difficulty has been en-

countered in designing a suitable commercial process for composting. The object then, is to set up experiments to investigate the relevant variables prerequisite to designing a satisfactory composting unit.

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I. REVIEW OF LITERATURE

Before any discussion of results and conclusions can be presented for examination, a background of the previous work in this field of composting is required. The review starts with the early developments of a compost pile, next accelerated methods, then the general bioxidation theory and proceeds to lead up to comparison of the present day processes of bio-chemical oxidation. In addition, there are chapters on analytical methods, organic waste complexity and coffee constituents.

A. Early Natural Methods

Prior to 1940, compost was mainly obtained from manure and plant refuse that had previously been biologically decomposed in heaps over a period of three to six months. This old practice in making compost heaps for the digestion of waste organic material has been more common in the British Isles (67) and Europe (67) than in this country. A variety of materials have been used in making compost including livestock excreta, weeds, cornstalks, grass, leaves, garbage and crop residues of all kinds. A school of gardening has developed around this practice which maintains that restoring fertility to the soil in the form of decomposed organic matter is much superior to the use of inorganic fertilizers. Probably the most celebrated advocate of this school is Louis Bromfield (6) in his articles about Malabar Farm.

Manure, which refers to the solid and liquid waste of livestock, was originally the only raw material used to prepare compost (86). It was then discovered (50) that other types of organic waste material could be used to prepare suitable synthetic manure for use as fertilizer. Crude composting piles consisted of moistening the waste and allowing it to rot over a long period of time. Experimental work by various organizations such as the agricultural stations in the country developed specific procedures for the building of compost heaps and their care. Additional studies showed that the rotting process can be speeded up by the use of nitrogen and phosphorus (45) containing chemicals. Commonly used compounds are ammonium sulphate, lime and super phosphate. Various patents (82) have been issued here and in Europe which cover the use of other chemical compounds which further accelerate the decomposition and raise the quality of the humus produced.

B. Developments Since 1940

Almost all of the scientific work and equipment designed on the process of composting has been done in the past sixteen years. As a consequence, little is known about the best procedures for composting of industrial waste. In the past eight years, considerable effort has been devoted to the fundamental concept of bioxidation and recently within the past two years, equipment has been developed on a pilot plant scale for the rapid decomposition of domestic garbage.

1. General. The term compost (2) is generally understood in America to mean a well decomposed mixture of manure, sods, leafmold, peat, or other organic matter and soil. It can also be defined to include decomposed organic waste from domestic garbage and food producing industries.

The process of composting which produces a fertilizer from organic matter accomplished two objectives:

1.) Concentrates the inorganic constituents such as nitrogen and phosphorus by reducing organic concentration through bioxidation.

2.) Conditions the waste through bioxidation so that when used as a fertilizer, it can be easily assimilated by the plants for continued growth.

Bioxidation of the waste is effected by micro-organisms which are mainly considered to be the aerobic type in the process of composting. Therefore, the literature search was largely devoted to methods employing these types of bacteria.

2. Accelerated methods. Many industrial plants such as the canning and meat-packing establishments have no method to dispose of their wastes except by (a) storage on waste or potential industrial land (69) (70), (b) discharging it into streams (39) (53), (c) discharging it into sewage systems of the locality (83). The organic content of these wastes is large and causes high pollution of the waters into which the wastes are discharged. Consequently, this creates a serious health and odor hazard to the community.

To dispose of the health hazard in industrial waste, the organic matter contained therein must be removed and decomposed partially or completely.

One of the most effective methods of treating organic industrial wastes is by aerobic oxidation. Of the several processes used such as biofiltration, lagooning and activated sludge, the latter is the most efficient for treating large volumes of waste (33). In this process, a mixture of waste and activated sludge is agitated and aerated (3). The activated sludge is a sludge floc produced from raw waste and contains zoogleal bacteria and other organisms. These floc growths hold waste materials while they are chemically or bio-chemically attacked.

This resultant organic sludge has been dried and sold as fertilizer. Milwaukee has been selling its organic sludge as milorganite for quite a while (41). A small plant (41) has been built by the Chicago Stockyards Composting Company for disposing of stockyard wastes and sewage sludge. The firm is contemplating constructing a new $250,000 plant. Other towns interested are; Cincinnati, Ohio; Bayside, N.Y.; Miami, Fla.; and Oakland Calif. (41).

A 1950 report (79) by U. S. Public Health Service states that the activated sludge process is used more than any other treatment in the bioxidation of domestic sewage. Hoover et al, (37) (38) have done considerable work with dairy wastes and report that half the organic waste is oxidized to carbon dioxide and water. Eckenfelder et al (15)

(17) have described pilot plant investigations of cannery waste treatment by the activated sludge process. Rudolph and Amberg (65) describe laboratory experiments on activated sludge treatment of concentrated paper mill white water. Coe (8) reports successful small-scale treatment of petroleum refinery wastes by the activated sludge method. Heukelekian (31) has evaluated the important factors affecting the extent of BOD reduction of strong penicillin and streptomycin wastes using non-flocculent growths. All of these applications give a reduction of BOD content in excess of 85%.

In 1954, it was revealed by Wagner College (46) of Staten Island that they have developed a process which turns garbage and refuse into rich plant food. At the same time, a similar bacterialogical conversion process has also been developed by Michigan State College. In this conversion process, metal, rags, paper and glass are separated from the garbage. The organic material left over is then carried to a grinder that reduces the waste to a fine subdivided state. The next and most vital part of the process is the digestor--a vessel with eighteen separate compartments in each of which the refuse is mixed and aerated and where the bacteria are used to decompose the refuse before it becomes fertilizer. Claims for this process are that it is odorless, smokeless, and the end product has an estimated value of $20.00 a ton commercially. No performance data is given.

Wiley (85) has investigated the variables of air rate,

agitation and moisture concentration in studies of high-rate composting of garbage and refuse. The investigations were made in six batch-type mechanical units having a working capacity of 11.1 gallons. He reports that agitation has little affect, optimum aeration rates in the range of 10 - 30 cubic feet per day per pound of volatile solids and an optimum moisture range of 55 - 69%. Loss in volatile solids over a six to nine day period average approximately 30%, indicating that the compost produced by aerobic thermophilic decomposition still contained decomposable organic matter.

Wiley states that about twenty-five runs were made in this study but only data for six of them are listed. He reports that the determinations of carbon dioxide and water correlate well with temperature but presents no figures or graphs to support this. Because of this correlation, the author concludes that temperature is a good measure of the degree of composting obtained and yet proceeds to use percent reduction of volatile solids as the basis for determining the amount of organic matter decomposed. This is inconsistent.

Temperature can be used as the criterion for degree of composting if it is related to the reduction in volatile solids or some other means of determining decomposition such as BOD or COD values. Carbon dioxide evolution is not a valid relationship since it is not only dependent on the rate of decomposition, but also on the amount of organic

waste present. Therefore, in using temperature as a measuring stick, it is necessary to know and keep constant all other test conditions except the variable being evaluated. This was not done.

This writer does not consider the results of Wiley's investigation with respect to air rate and moisture content to be conclusive. Considering high air rate evaluation, Wiley states that the temperature dropped due to cooling and dehydration of the organic waste and hence these particular rates will not produce a good compost. Water is required by bacteria to decompose organic waste thereby creating heat. The moisture content should have been constant during these runs. In high moisture content experiments, the temperature also dropped due to reduced organic concentration resulting in less heat evolved which in turn heated a larger quantity of water. A corresponding per cent volatile solids reduction on each run would have helped to clarify the situation.

For the optimum values given, Wiley does not attempt to support them with any theoretical suppositions concerning bioxidation.

3. Patented composting equipment. There is no equipment on the market at the present time specifically designed for composting of organic waste that is commercially feasible. However, there are several patents on composting apparatus.

In 1939, a patent (13) was issued Earp-Thomas for a di-

gestor design. The organic waste was decomposed in a tank with superposed decks. The organic refuse, broken up into small particles, enters the top where it is agitated on the decks by a plurality of mixing arms which also move the material from one deck to the other. Prior to digestion, the organic waste is mixed with culture suitable for aerobic decomposition. The temperature in this apparatus was reported to vary from 80° to 180° F.

In 1950, Earp-Thomas (14) designed another digestor called an activated composter. The composter was a large chamber of square cross-sections. Each side is constructed of multiple flaps similar in appearance to a venetian blind. The angle of these flaps is adjusted in accordance with the amount of air desired. Again, the organic waste being treated is inocculated with aerobic bacteria selected from decaying organic matter. Addition of chemicals such as lime, ammonium sulfate, and calcium nitrate is optional. The temperature, which varies over a range of 80° to 140° F., is regulated by the amount of air which is passed through the waste.

In 1952, Taylor, Voegtlin and Schlosser (77) patented a digestor for the manufacture of fertilizer which consisted of a tank with superposed decks similar to that of Earp-Thomas'. Agitator arms remain stationary while decks are moved by a central shaft. No temperature conditions, pH values or air rates are given.

All of the foregoing designs are not practical, as the waste is poorly agitated and the aeration is inadequate.

In addition, the equipment is expensive and the mechanical complications are considerable. The following design seems to be the most logical and economical approach so far, but it still does not effect a completely satisfactory compost.

Eweson (21), in 1952, was given a patent for an apparatus for making organic fertilizer. This unit was developed for the biological decomposition of moist organic matter through the propagation of aerobic bacteria. The apparatus comprised of a tank having an agitator, means for forcing air under pressure into the tank at the bottom level and a plurality of outlet conduits located at various levels within said tank for withdrawing spent air and generated gases. The organic waste treated was first ground to a uniform fine condition, inocculated with a seed stock of aerobic soil bacteria, mixed with certain nutrients and minerals such as nitrates, urea, ammonium salts, phosphates and lime. Temperature varies between 90° to 150° F. depending on the stage of fermentation. Quantity of preheated air used is one-half cubic feet per cubic feet of material treated.

4. Economics and design of equipment. No composting design data was found in the literature. All published data is concerned with the activated sludge process. Specific studies of agitation and aeration have been conducted.

Eckenfelder and Moore (19) have shown how pilot plant data can be utilized for full scale design. Cost of constructing bioxidation plants of various sizes also is presented.

Oldhue (52) has studied the theory and design of mixers

for aeration of waste. De Beeze and Liebmann (10) have done considerable work in the field of aeration. Eckenfelder (16) discusses the power costs obtained with varying air rate.

C. Bioxidation of Organic Matter

1. Theory. It has been stated by various investigators (45) (2) (27) (67) (85) that the organic matter in the composting process is broken down by the action of bacteria and micro-organisms. This breakdown by bacteria is known as bioxidation. Bacteria (24) has been defined as microscopic unicellulor plants, possessing no well defined nucleus, devoid of chlorophyl, and reproducing by binary fission. Changes brought about by bacteria in their environment are a result of their life activity, which is collectively referred to as metabolism. Metabolism (48) involves the intake, digestion, and assimilation of food into tissues, and the transformation of the potential energy of the food into kinetic energy with which the organisms accomplish work, together with the elimination of any waste products formed. Metabolic processes may be divided into two catagories called catabolism and anabolism. The catabolic changes are exothermic and the anabolic changes are endothermic. The catabolic process takes place after the food enters the cell and the material is broken down with the liberation of energy. Where the food is of such a nature that it cannot diffuse and enter, the cell bacteria excrete enzymes to hydrolyze it and con-

vert it to more readily diffusible form. The hydrolytic reactions taking place outside the cell by means of exoenzymes (59) liberate only small amounts of energy which in no way can be made use of directly in the cellular metabolism. Metabolism starts after the food materials enter the cell. The materials inside the cell are broken down by means of endoenzymes with the liberation of energy. Certain fractions of the catabolic products are then used as food for the anabolic processes of the bacteria, building up their cellular materials by utilizing the energy liberated in the catabolic processes.

Catabolic dissimulation (26) of food such as organic wastes may take place either under anaerobic or aerobic conditions. Energy liberation under both conditions is the result of a series of steps involving the dehydrogenation of the organic materials. Atmospheric oxygen (24) serves as the main hydrogen acceptor under aerobic conditions whereas nitrates, sulfates and carbon dioxide serve as hydrogen acceptors under anaerobic conditions.

Under anaerobic conditions (32), the food materials are broken down incompletely to various intermediate compounds in which most of the energy of the initial material still remains. Various organic acids such as acetic, butyric, propionic, valeric, lactic as well as lower alcohols-ethyl, butyl, propyl, etc., are formed from carbohydrates, fats and proteins. The products of the degradation of nitrogenous materials are amino acids and ammonia. The gases formed are

primarily methane, hydrogen, carbon dioxide and hydrogen sulfide.

Under aerobic conditions, the decomposition of food materials is complete, all the energy locked up in the food materials is liberated resulting in greater bacterial growth. The end products are carbon dioxide, water, ammonia, and nitrates, none of which create offensive conditions.

2. Factors affecting aerobic microbiological oxidation of organic waste.

a.) Food.(32) Chemical composition and the physical state of subdivision of the organic waste will affect its availability to the bacteria. Some organic materials are more easily hydrolyzed and diffuse more readily than others. Example--sugar is hydrolyzed more quickly by enzymes than lignin.(24) As complexity increases, the availability decreases. Oxidation of the insoluble fraction of the food, which is governed by its physical state of subdivision (46), is controlled by the rate at which it is hydrolyzed and diffused through the cell membrane. The carbon-nitrogen (32) ratios of the organic materials also affects the rate availability. Organic material, entirely devoid of nitrogen such as sugars, is available to only a few bacteria despite its fine state of subdivision. Unless nitrogen is present in sufficient quantities in the surrounding medium, such a substrate is not oxidizable, Organic nitrogen (24) is broken down in a series of stages, to ammonia nitrogen before it becomes available for structural purposes during the

oxidation of carbonaceous materials. For the rapid oxidation of carbonaceous materials sufficient nitrogen must be furnished to supply the cellular needs of the micro-organisms. Investigators (32) (30) (72) have shown that one part of nitrogen will be required for every twenty parts BOD. Phosphorus also is essential for cell nutrition and oxidation of organic matter. If a waste is deficient in this element, its addition accelerates the rate of oxidation. Amount (32) (30) (72) required is one part of phosphorus to 75 - 100 parts of BOD.

b.) Organisms.(31) In the biological treatment of wastes the changes desired can be effected by the number and type of organisms that the waste is initially seeded with. The greater the initial number of organisms the shorter the lag period will be and hence the greater the over-all rate of oxidation. As the number of organisms are increased, the rate of oxidation is increased to a certain point, beyond which the additional organisms do not give an increase in rate. Processes of acclimatizations (24), training, adaptations, selection and mutation may be used to develop within limits the optimum number and type of organisms necessary for the maximum rate of oxidation.

c,) Environmental conditions.(32) Micro-organisms induced into the waste to initiate decomposition can be of no measurable value unless the conditions under which they exist are favorable to growth. Micro-organisms are affected by

water, pH, temperature, oxygen, light, agitation, pressure, foaming and inhibiting materials.

1. Actively growing micro-organisms (32) are composed of from 75 to 95 per cent water, and therefore, water is indispensable in their metabolism. Both the shape and size of many organisms depend upon the absorption of water and the setting up of sufficient internal pressure through osmosis to keep the cell extended. Without water, the cell will collapse and a condition comparable to a wilted plant will exist.

2. The rate of oxidation does not seem to be so critically affected by departures from the optimum pH of 7 (11) (68). Between values of 6 and 8, the oxidation rate is not materially affected.

3. Micro-organisms (24) (32) (71), like higher plants, can grow and carry on their life activities at the optimum rate only within a rather limited temperature range. This is spoken of as the optimum temperature of the organisms, a term which is intended to convey the idea that at that particular temperature life processes, as a whole, are functioning best for the welfare of the organism in question. The lowest and highest temperatures at which an organism will grow are spoken of as its minimum and maximum temperatures respectively. If the optimum is relatively low, around 68° F. or less, the organisms is said to be phychrophilic (cold-loving). If the optimum lies around 113° F. or above, the organism is said to be thermophilic (heat-loving). Those

organisms with an optimum temperature between 65° to 113° F. are said to be mesophilic (medium temperature loving). The higher the temperature, the more active micro-organisms become, provided the temperature or some secondary effect (32) does not become a limiting factor. Therefore, the rapidity of growth and general metabolic activity of thermophilic micro-organisms should produce the maximum oxidation rate.

4. Appreciable quantities of oxygen (24) (32) are needed in the microbial synthesis of organic matter since half the dry weight of the micro-organisms is oxygen. Two groups of micro-organisms, aerobe and facultative organisms, can be used in the aerobic process.

The oxygen rate varies from (31) (21) 1.0 to 3.0 cubic feet of air per pound of bio-chemical oxygen demand. Aeration of the waste is accomplished by passing bubbles of suitable size through the entire medium. A smaller quantity of air is required when the bubbles used are of small size, but it costs more to do this (10).

5. Light (24) is injurious to many if not all nonphotosynthetic micro-organisms and would therefore be harmful to the organisms produced in the aerobic process.

6. Use of pressure (24) in tanks has been employed to increase the effectiveness of aeration. Pressures from 20 to 40 psi have been used in some processes.

7. Effective agitation (4) (52) of the wastes increases the efficiency of aeration and consequently, decomposition. Gentle agitation is beneficial to the growth of most micro-

organisms, while vigorous agitation and vibration have been reported as detrimental and destructive if prolonged for a sufficient period of time.

8. Excessive foaming (74) (59) is the result of aeration and gas products of the waste being treated. Decreasing the foam increases oxidation rates. The type of chemical agent used to prevent excessive foaming is selected on the basis of its inertness to the micro-organisms developed.

9. Certain groups (47) (65) of chemicals are poison to a particular class of organisms. A knowledge of the general components of the waste will further aid in determining the optimum types of micro-organisms with which to seed the wastes.

D. Reducing the Complexity of Organic Waste

In subjecting coffee waste to biological oxidation by the aerobic process, the chemical composition and physical state of subdivision of the waste will determine to a large extent its availability to bacterial decomposition and consequently the time necessary for composting. It is therefore evident that simple organic materials are more easily hydrolyzed and diffused more readily than others as in the case where sugar is hydrolyzed more quickly by enzymes than is lignin (24). Since the complexity of coffee waste is known to be great, the task of simplification must be evaluated in order to effect a decomposition rate that is practical. A variety of methods may be used for converting complex carbohydrates to comparatively simple compounds (61). These

methods may be classified into two catagories which are;

1. Cooking the waste to be treated prior to enzymatic action by mold bran and fungal amylases.

2. Also cooking the mash subsequent to acid hydrolysis using hydrochloric or sulfuric acid.

The above processes which convert celluloses and other carbohydrates such as proteins to sugars and hydrolyzed products are saccharification methods.

Cooking of the waste can be accomplished by a batch or continuous method at temperatures of 120° to 310° F. and under pressure for a specific time. A most unique method of cooking has been developed by Unger (78) who mixes the waste with steam at 350° to 365° F. in a Schutte-Koerting jet heater for a period of 60 seconds. This process, which is continuous, is employed in the plant of Joseph E. Seagram & Sons, Inc., Louisville, Ky. The optimum practical pH range is 5.4 to 5.6.

Final hydrolysis or saccharification of the waste is accomplished by dilute acid or biological action at various temperatures, pressure, dilutions, and times of contact, as reported by numerous investigators (12) (84) (29) (73). In the case of coffee hydrolysis, time and economics are of the essence. With these factors as a guide, the hydrolysis of wood and agricultural residues were looked for in literature (57) (28) (43). Practically all the articles surveyed employ the process of hydrolysis to produce fermentable sugars for alcohol or yeast production. Although it is con-

ceivable that some of these methods could be applied in resolving the coffee waste problem, none of the methods invented are utilized on a commercial basis. However, according to an I & E CHEM., saccharifications report titled "Cheaper Sugar from Wood"(78), it is stated that the Rheinau process used in Germany to produce sugars from waste wood is feasible. The process consists of two steps; (1.) from chipped wood, the easily hydrolyzable pentosans are taken out by boiling in a kettle with 1% hydrochloric acid under pressure and (2.) the resulting wood residue containing cellulose and lignin is dried and subjected to the main hydrolytic action by continuous countercurrent treatment with 41% hydrochloric acid at 20° C.

This method boasts a yield of 31% dextrose, 22% of prehydrolyzate sugar, 8% of sugar in the mother liquor from the dextrose, and 30% lignin based on the dry waste.

In applying this method to the hydrolysis of coffee, the main consideration is not in the type of compounds produced, but rather the degree of simplification or hydrolysis effected prior to composting. However, if this method manufactures from coffee waste, products which can be utilized in other fields on a more profitable basis other than composting, these outlets should be investigated.

E. Aerobic Versus Anaerobic Methods

As mentioned in previous chapters, all composting investigators and equipment designers used aerobic bioxidation

in attempting to effect a suitable compost from organic waste. When the results of this study showed that poor decomposition of coffee waste was effected by the aerobic method, it was decided to consult the literature further to see if some factors had been overlooked and if there were other methods more suitable such as the anaerobic process.

Gurnham (27), in his new book concerning principles of industrial waste treatment, gives the following requirements of the aerobic versus the anaerobic bioxidation methods:

	Anaerobic	Aerobic
Main Purpose:	Bioxidation of organic matter	Clarification of sewage by absorption and oxidation of organic waste
Products:	60 - 75% methane 40% of original wt. in humus form	CO_2, H_2O, nitrates 70% of original wt.
Max. Conc. of BOD handled:	200,000 ppm. and up	2500 ppm. (max.)
Max. Conc. of solids:	200,000 ppm. and up	3000 ppm. (max.)
Equipment:	-	more expensive than anaerobic
Space requirement:	-	more extensive than anaerobic
Objection:	Offensive odor	--

The one bad feature of the anaerobic method is the strong odors associated with it. These odors may be controlled by careful selection of micro-organisms.

Eckenfelder and O'Connor (20) have reported that 29% of the BOD of a cannery waste was directly oxidized and the remainder went into synthesis of new sludge using aerobic

oxidation. Wiley (85) states that the aerobic, thermophilic bioxidation of garbage and refuse gives an average decomposition of 30%.

Pearson, Feuerstein and Onodera (54) used the anaerobic process in the treatment of winery wastes and listed a 70% reduction of organic content as measured by volatile solids. Boruff (5) and Buswell & Hatfield (7) have done a considerable amount of work with anaerobic fermentations. Jacobs (41) advises that concentrated wastes high in BOD can be more economically treated by anaerobic means than by aerobic treatment. He further states that experiments with paper mill wastes have shown great promise for anaerobic decomposition and that any organic waste resulting from the manufacture of foodstuffs should be susceptable to this treatment. Gaden (23), in a recent article, gives the chemical engineer a better concept of the tools of fermentation.

Because of the foregoing information, this writer believes that the anaerobic process is ideally suited to the composting of coffee waste. Properly controlled, it will effect a gaseous product readily usable as fuel and a humus-like solid which can be sold for soil-conditioning. In addition, since coffee waste is considered uniform from batch to batch, possibilities for various other products are unlimited.

F. Analytical Methods

In any study concerning bioxidation, the decomposition

rate of organic waste must be determined at periodic stages of the process in order to ascertain the effects of the variables evaluated.

Of all the composting papers published only one, Wiley's (85) lists a method for determining the amount of decomposition. He employs the volatile solids procedure to determine the overall percent oxidized at the end of a run. It was not used to show the decomposition rate at various stages during the run.

The activated sludge process almost exclusively uses the bio-chemical oxygen demand procedure as standardized by the U. S. Public Health Service (74). BOD values for 5 day or 20 days incubation can be obtained depending on the extent of organic waste present.

These long incubation times for BOD determinations created a demand for shorter tests which resulted in the development of chemical oxidation methods. These procedures used chemical reagents to determine the oxygen demand of the organic waste and hence give values designated COD, chemical oxygen demand.

One group of researchers directed their effects toward the use of dichromate as the oxidizing agent. In 1926, Adeney & Dawson (1) and in 1927, Von Fellenberg (22) used potassium dichromate and sulfuric acid for determination of organic matter. Rhame (62), in 1947, and Ingols & Murray (40), in 1948, developed similar procedures but modified them so that they were easier to carry out. A further alteration

has been devised by Moore, Kroner and Ruchhoft (49). In 1950, the U. S. Public Health Service (75) came out with a dichromate method to replace their previous permanganate tests. The most rapid test for determining organic pollutions was invented by Pepinsky, Porges and Hoover (55) in 1951.

The other group consisting solely of Johnson, Halverson and Tsuchiya (42) have developed a technique using iodic acid for the measurement of the complete oxygen demand.

Roberts and Sanderson (63) showed in comparison tests among the various methods except that of Pepinsky et al, that the best procedure, with regard to results obtained and reproducibility, is the U. S. Public Health Service (75) method. This method, they state, uses comparatively simple equipment and each determination has an elapsed time of three hours.

The rapid oxygen demand test developed by Pepinsky et al, (55) is carried out in a 500 ml. Phillips pyrex beaker without transfer of the solution in an elapsed time of twenty minutes. Its advantages, as stated by the inventors, are reproducibility, speed and simplicity of equipment and operation.

In taking oxidation measurements periodically of coffee waste undergoing composting, it is not necessary to obtain precise results and therefore, it was decided to employ the rapid test of Pepinsky et al (55) solely on the basis of reproducibility and speed of operation. This test

was modified because of the high concentration of coffee waste involved which increased the time of testing to ninety minutes.

The oxygen demand of waste determined by the above test is supposedly equivalent to a 20-day bio-chemical oxygen demand value. However, Kaufman (44), in his comparison studies of BOD and COD, states that the relationship is not valid for all wastes because of the specific conditions peculiar to each.

The most rapid measure of the oxidizing or reducing strength of an organic waste mixture is provided by the oxidation-reduction potential, ORP, also known as the redox potential. This potential value is developed electrically because reducing agents have a tendency to release electrons and oxidizers are electron acceptors. Positive values indicate aerobic bioxidation and negative figures indicate anaerobic.

Rohlich (64) and Hood (34) have commented on the use of ORP measurements in sewage treatment. Eckenfelder and Hood (18) have used ORP as a process control measurement in the bioxidation of cannery wastes. Difficulties of instrumentation and measurement have hindered the development of this method.

The nitrogen and phosphorus contents of coffee waste were determined by official methods of analysis of the Association of Official Agricultural Chemists (51). Also obtained from this reference were procedures for measurement of ash; oil, fats and waxes; crude fibre; and protein.

G. Composition and Chemistry of Waste from Powdered Coffee Production

The coffee waste is probably composed of the following general ingredients (80); ash; oils, fats and waxes; protein and crude fibre.

1. Composition of coffee waste ash (1 to 10% concentration). The ash represent inorganic mineral ingredients of the waste and is therefore a mixture of many salts. These are the salts which are essential in the germination of the coffee plant. Chemical examination of the ash indicated the presence of potassium, sodium, calcium, magnesium, iron and probably other basic elements. These metals are combined more or less completely with phosphorus and sulfur to form their respective salts.

2. Composition of oils, fats and waxes (20 to 30% concentration). Many investigators have studied the foregoing constituents of coffee since 1837. The numerous results reported agree roughly as to their physical and chemical constants, but differ widely as to the amounts of the various acid components present. These organic factions exists in mixtures of great complexity. They are of such a character that they can be split in part by action of heat or roughly separated by different solvents so that analysis yields a series of products which represent the cleavage products of a cracking process. The ready splitting of the fats and waxes by heat indicates they may be considerably modified by high temperature and pressure or the equivalent.

The fat extracted from coffee contains a large amount of unsaponifiable matter. The principle acids reported by investigators are; linoleic, oleic, palmitic, carnaubic, stearic, capric, and an unsaturated hydroxy acid. Since the literature survey of this subject was limited to the essentials, there are undoubtedly other acids present in addition to those listed here. Organic glycerides and peroxides have been found in coffee in small amounts.

3. Composition of protein (10 to 20% concentration). Nitrogenous compounds common to other plant waste have been found in coffee such as gluten, etc. No special proteins peculiar to coffee have been found in literature although, undoubtedly, some exist. During exposure to heat, oxidation reduction of the protein substances occur resulting in partial cleavage and subsequent hydrolysis.

4. Composition of crude fibre (40 to 70% concentration). The fibrous portion of the coffee waste contains many forms of cellulose, pentose or pentosans, galactose, glucose, and gluocosides, and starch. Upon exposure to heat, cellulose, sugars, and starches are partially carmelized and darkened and some of them, such as pentosans, yield small quantities of furfuraldehyde and other products. The furfuraldehyde may be reduced at once to furfuryl alcohol and some CO_2 to carbon monoxide. Esterification of furfuryl alcohol with aliphatic acids may take place. Woody constituents made up of cellulose addition products and modifications undergo

splitting and are partially carbonized.

5. Other constituents. Investigators have also found these following organic compounds not mentioned in the foregoing sections; higher fatty acids, acetic acid, hydroquinone, methylamine, pyrrol, acetone, pyridine, ammonia, trimethylamine, formic acid, isovaleric acid, and methyl ether of saligenin, which is claimed to be the principal constituent of coffee oil. Formic acid and hydroquinone have an inhibiting affect on micro-organisms.

II. EQUIPMENT

Coffee waste was to be composted by aerobic microbiological oxidation in an apparatus called a digestor. This piece of equipment was designed to treat a gallon of liquid slurry and the material of construction is pyrex glass. The column, which is jacketed, has a 3-inch inside diameter and is 36 inches in length. The bottom of the column is fitted with a rubber stopper containing a thermometer, air sparger and sample tube valve. Details of the digestor and bottom fixture are shown in figures 1 and 2, respectively.

Hot water from a constant temperature bath is circulated through the jacket of the column by means of a centrifugal pump and returned. The heating wire coil on the column is connected in series with the mercury actuated temperature control circuit of the hot water bath. The temperature of the bath is adjusted by the reservoir of mercury in the off-

on control tube.

Air is supplied by a compressor and is regulated by a flowmeter to the digestor. The air is first preheated in a coil immersed in the water bath. Steady flow conditions are maintained by a reserve tank and pressure regulator.

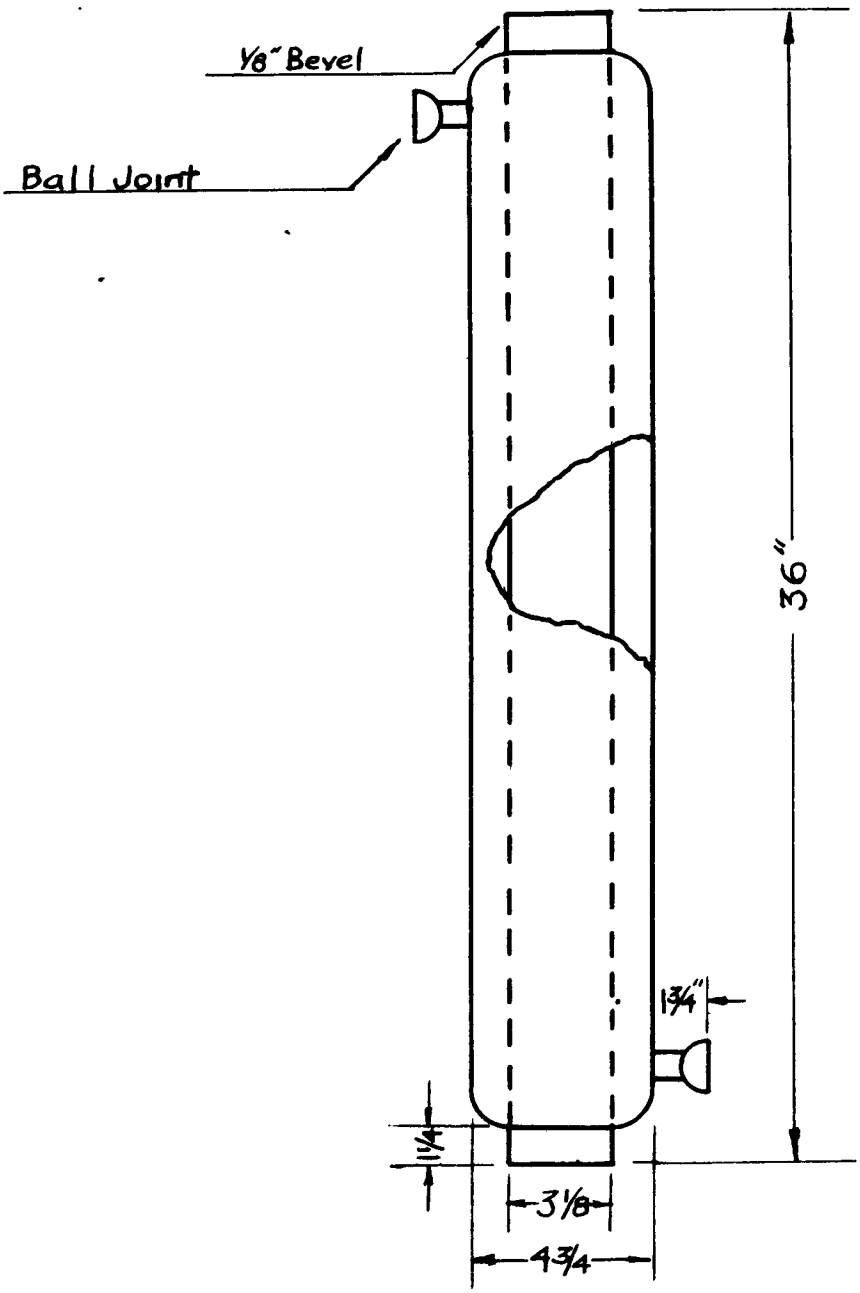
The coffee waste was ground in a comminuting machine through a triple zero perforated screen.

Figure 3 is a flow-sheet of the composting unit and a picture (figure 3A) shows how the equipment was arranged for operation.

Specifications for all experimental and analytical equipment are listed in appendix B, page 74.

FIGURE 1

DIGESTOR DESIGN



SCALE
1/6" = 1"

FIGURE 2

BOTTOM FIXTURE

AGITATOR

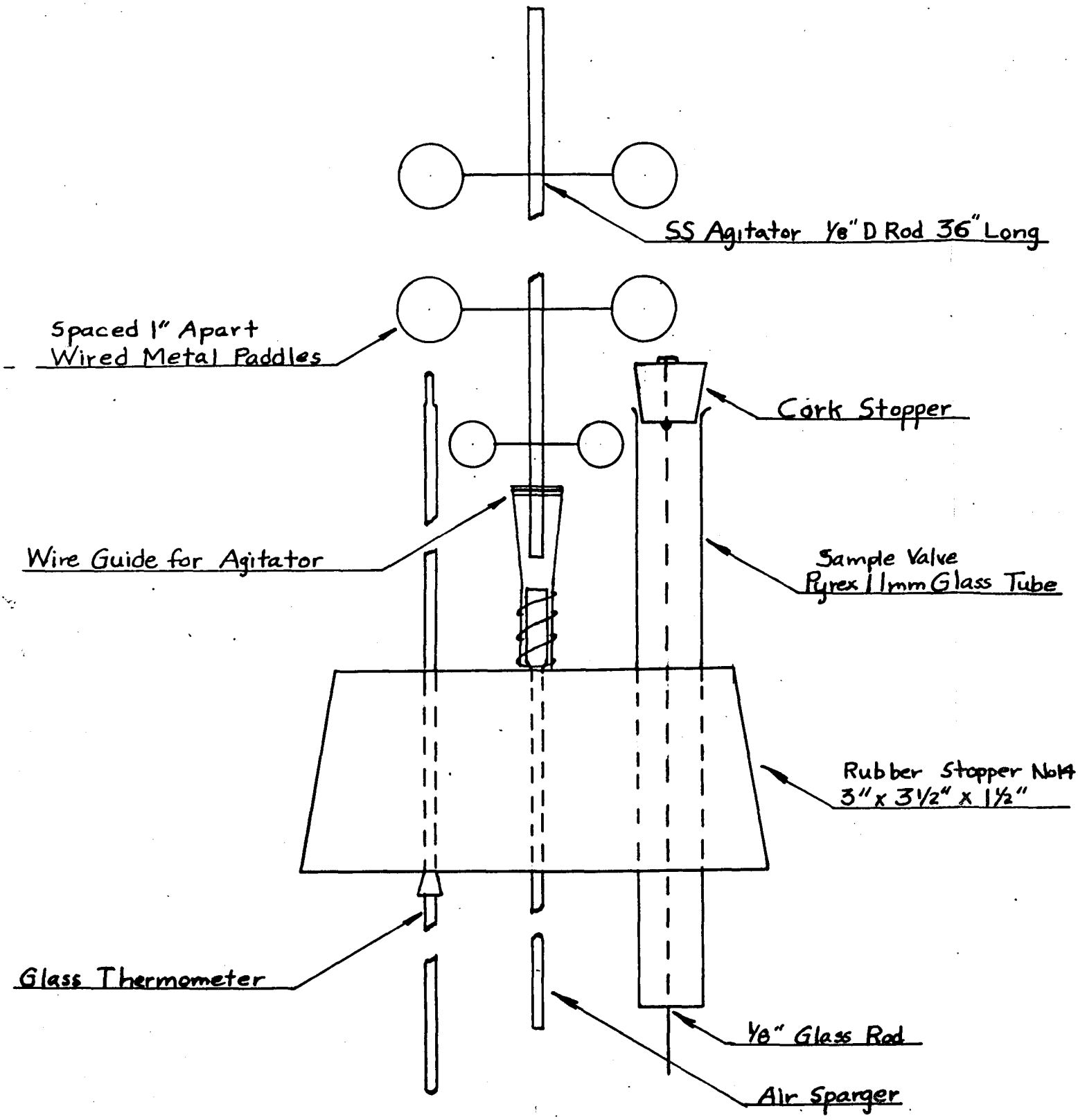


FIGURE 3

COMPOSTING FLOWSHEET

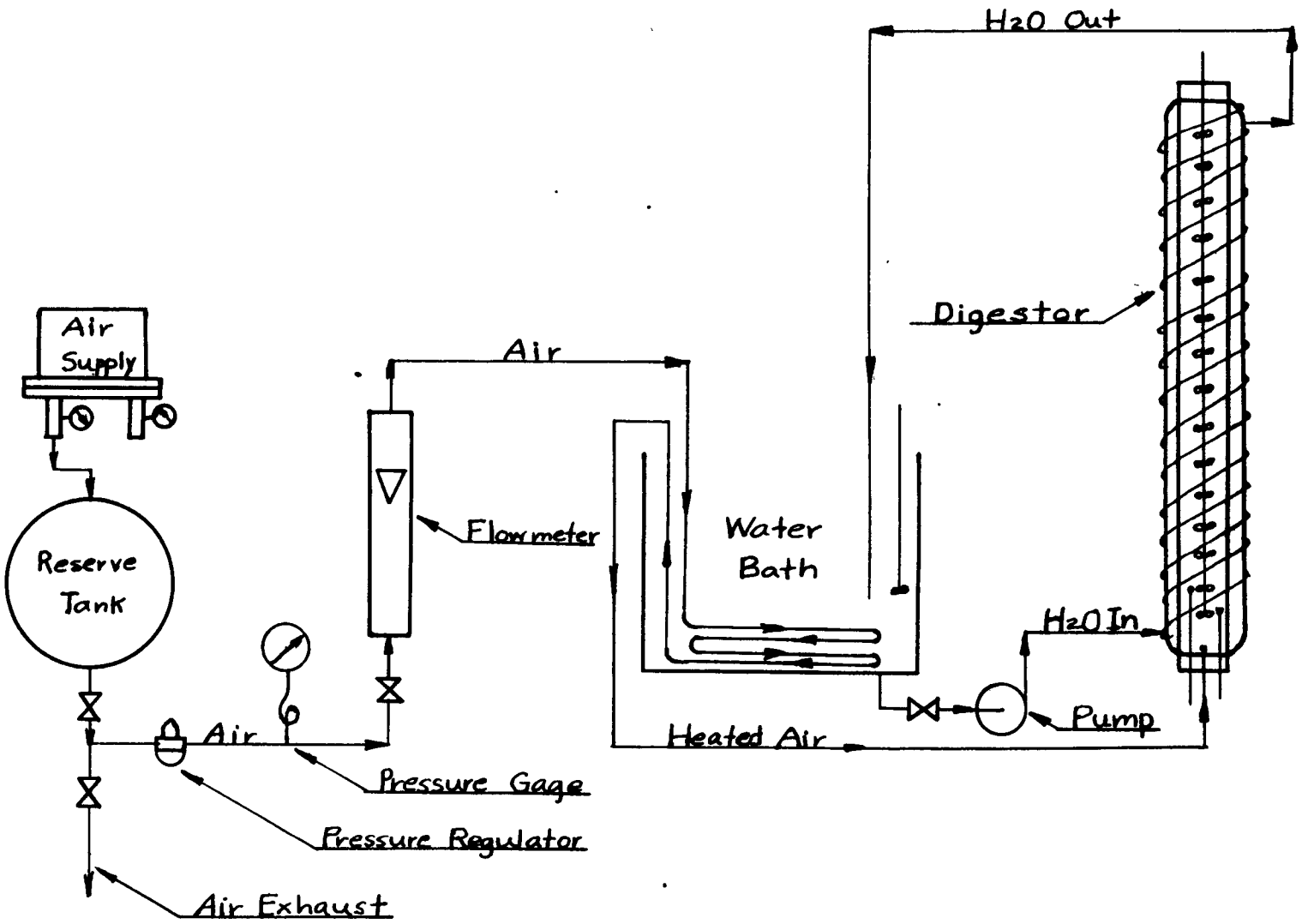


FIGURE 3A
PICTURE of COMPOSTING UNIT

III. PHYSICAL PROPERTIES OF COFFEE WASTE

The general composition of coffee waste as received from Maxwell House Division is as follows on a dry basis: (80)

Percent Ash	Percent Oils Fats & Waxes	Percent Protein	Percent Crude Fibre
0.3	20.6	11.3	67.8

The nitrogen and phosphorus concentrations of the waste are 1.8% and 0.1% respectively (51).

The chemical oxygen demand, as determined by the rapid test developed by Pepinsky et al (55), is 1.29 grams of oxygen per gram of untreated coffee waste. On autoclaved waste this value is increased to 1.74 due to the almost complete hydrolysis effected and hence determination of the true COD value. Therefore, the COD value of the raw waste is estimated to be only 74% of the true figure.

Hoover, Jasewicz, Pepinsky and Porges (55) list 1.25 grams of COD per gram of sludge from the activated sludge process.

IV. EXPERIMENTAL PROCEDURE

A. Preparation of Active Seed Material

To initiate the decomposition of the coffee waste it was necessary to seed it with micro-organisms which had to be developed with the waste in question. This was accomplished by saturating with water a mixture of 30% topsoil, 30% humus and 40% coffee waste. This mixture was allowed to ferment at a temperature of 130° F. for a period of seven days. At the end of this time, the fermented mud-like mass

was diluted to 15% solids and aerated in the digestor for five days at a temperature of 130° F. The resulting compost was refrigerated and used periodically as a seed material for the raw coffee waste.

B. Preparation for a Composting Run

The coffee waste is first ground and then mixed with a predetermined amount of activated compost. Water is added to dilute the mixture to a solid concentration of approximately 15%. This resultant aqueous slurry was analyzed for COD value. Based on this figure, nitrogen was added in the amount of one part to twenty parts of COD less the amount present in the waste. The nitrogen was supplied from a mixture of ammonium sulfate and ammonium nitrate. Phosphorus was added in the amount of one part to seventy-five parts of COD less the amount present in the waste. Phosphorus was supplied as ortho-phosphoric acid. The pH was adjusted to approximately 7.0 by the use of calcium oxide.

This prepared mixture was charged to the digestor. The agitator was turned on and the contents were heated to the desired temperature by the warm water in the jacket. When the desired temperature was attained, the run was commenced by passing a measured rate of preheated air through the inoculated mass. On the average of every three hours, a sample was taken and analyzed for pH value, solids concentration and COD content. This was repeated until a total of

twelve hours had elapsed at which time the run was halted. The contents of the digestor were removed and it was cleaned out. Runs were repeated according to the variables studied using freshly prepared mixtures each time.

C. Conditioning of Coffee Waste Through Autoclaving

It was reasoned that if the organic complexity of coffee waste was reduced, increased decomposition rates would be achieved. To prepare waste for this type of evaluation, it was subjected to a treatment developed by Unger (81). In this treatment, the waste is mixed with steam at 350° to 365° F. in a Schutte-Koerting jet heater for a period of 60 seconds. To simulate these conditions, moist coffee waste was placed in a two-inch steel bomb and heated by an oil bath to 365° F. for a period of five minutes. The resultant material, which had a strong, sweet, caramel odor, was ground and prepared as usual for a composting run.

V. CALCULATIONS

A. Air Rate

The Fisher flowmeter reads directly in liters per minute under standard conditions of 14.7 pounds per square inch absolute pressure and 20° C. temperature. Since standard conditions did not exist, this value had to be corrected according to the specific gravity of the gas, temperature and pressure of the system. Figure 4, is a nomograph for this flowmeter which is used as follows:

1.) With a straight edge, connect the temperature (T)

with the absolute pressure (P) and note the point where it intersects with the reference line (R).

2.) Move the straight edge to connect this point of intersection with the specific gravity (G) of the gas used and read the correction factor at the point where the straight edge intersects the correction factor line (K). Specific gravity of air is taken as 1.0 at standard conditions.

Assuming 2.0 liters per minute at 2 psig. and 85° F. the nomograph correction factor is 1.05. The corrected air flow is 2.1 liters per minute. This value was then converted to cubic feet per pound of COD per hour:

$$= 2.1 \text{ liters/min.} \times \frac{454 \text{ g. per pound}}{547 \text{ g. COD required}} \times 28.3 \text{ liters/cu.ft.} \times 60 \text{ min/hr.}$$

$$= 3.69 \text{ cu. ft./lb. COD/hr.}$$

B. Chemical Oxygen Demand (COD) (Appendix A, Page 70)

In order to determine the COD (55), the weight of sample and corresponding milliliters of thiosulfate had to known. Also, a blank determination in milliliters of thiosulfate was essential. With this information, the COD was calculated according to the following formula:

COD per gr. of slurry

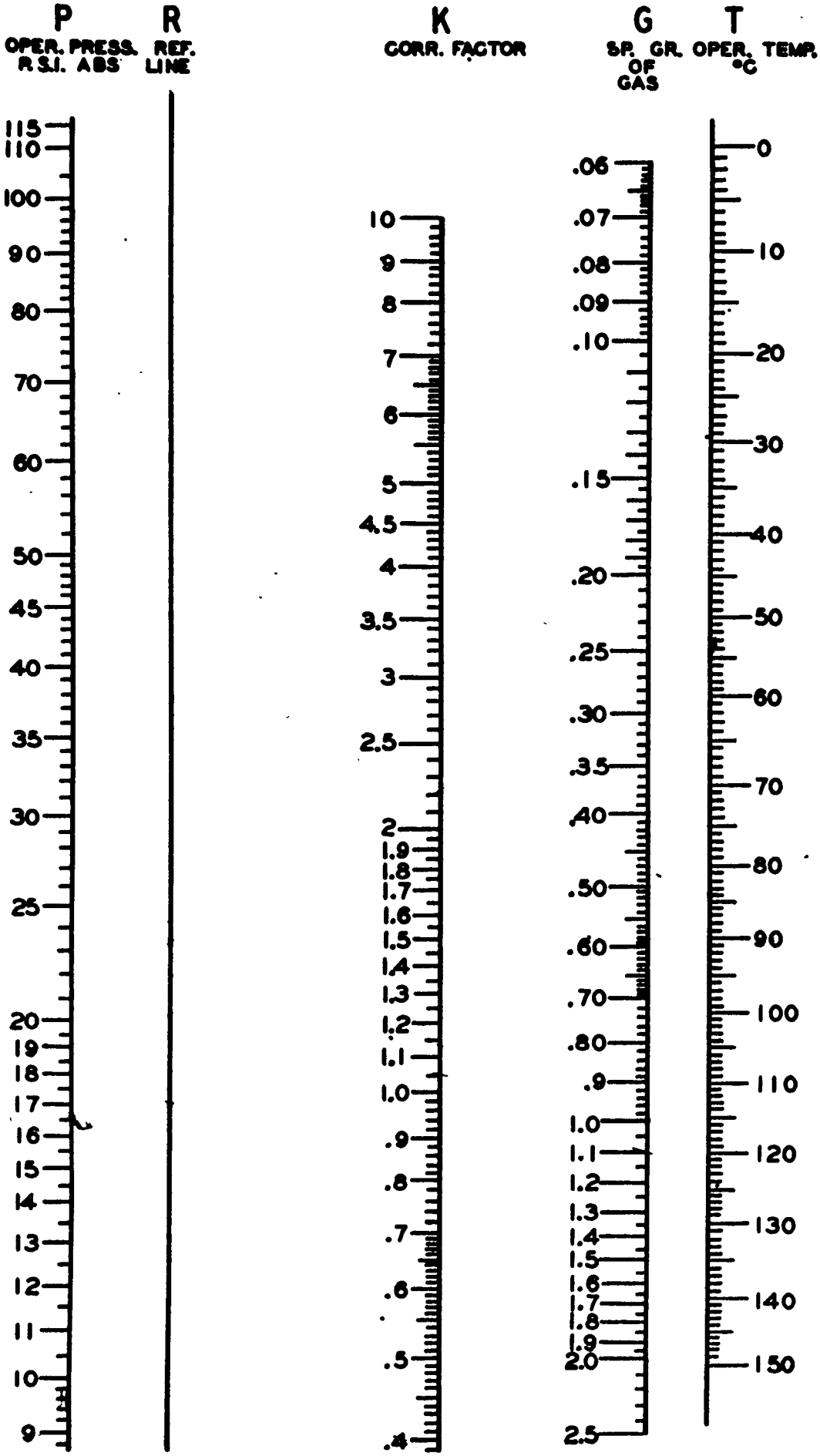
$$= \frac{(10)(\text{ml } Na_2S_2O_3 \text{ in blank} - \text{ml } Na_2S_2O_3 \text{ in sample})(N)(8)}{(1000)\ (\text{Wt. of sample})}$$

For example, assume a sample weight of 1.365 and 17.6 of thiosulfate to reduce the excess dichromate. A blank gave 43.35 ml. of thiosulfate to reduce dichromate.

$$\text{COD per gr. of slurry} = \frac{(10)(43.35 - 17.6)(0.1073 \text{ normality})(8)}{(1000)\ (1.365)}$$

COD per gr. of slurry = 0.168 g. of oxygen needed.

FIGURE 4



Nomograph for Fisher Laboratory Flow Meter

However, this value was taken at 16.5% solids while all the other determinations were made at a base solids of 15.3%. This original figure had to be corrected to the base solids. The corrected COD is:

corrected - COD= 0.168 x $\frac{15.3}{16.5}$ = 0.156 per gr. of slurry

Cumulative reduction of COD (corrected) was calculated in percent.

C. Determination of Optimum Composting Conditions

The percent COD results were plotted against the elapsed time on regular graph paper. All runs, pertaining to the same variable, were plotted on one sheet. These families of graphs were then studied to select the optimum range of the variable investigated.

VI. TABLES AND GRAPHS

A. Data Tabulations

TABLE I

EXPERIMENT 1

Variable - Air Rate

Air Rate Investigated - 0.9 cu. ft/lb.COD/Hr.

TIME	pH	AIR RATE	SOLIDS WEIGHTS			WT. OF COD SAMPLE	ML. OF $Na_2S_2O_3$ REQUIRED
			After	Before	Tare		
12:30 P.M.	6.6	0.5	3.5	12.36	1.66	1.267	19.75
3:30 P.M.	7.3	0.5	3.265	11.440	1.620	1.30	20.50
7:45 P.M.	7.15	0.5	3.215	11.210	1.610	1.045	22.75
9:30 P.M.	7.05	0.5	3.320	12.050	1.605	1.305	19.60
12:30 A.M.	6.9	0.5	3.015	10.090	1.610	1.375	18.50

Approximate Temp.	--	130°F
Ratio of raw to active waste	--	4.1
Original pH	--	5.5
Grams of CaO to raise pH	--	110
Air gage pressure	--	1.5 psig.

Amt. of waste treated	3406 g
Total oxygen required	547 g
G. of $(NH_4)_2SO_4$ added	46.4
G. of Phosphoric acid added	30.7
Time of run	12 hours
Ml. of Thio for blank	43.35
Agitation rate (#1)	150 RPM.
G. of NH_4NO_3	31.4

TABLE II

EXPERIMENT 2

Variable - Air Rate

Air Rate Investigated - 1.8 cu. ft/lb. COD/Hr.

TIME	pH	AIR RATE	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S203 REQUIRED
6:00 A.M.	6.8	1.0	2.770	9.970	1.615	1.180	23.4
9:00 A.M.	7.45	1.0	3.21	12.28	1.61	0.995	20.7
12:00 P.M.	7.10	1.0	2.84	9.96	1.63	1.160	22.4
6:00 P.M.	6.9	1.0	2.550	7.870	1.645	1.150	21.5

Approximate Temp. -- 130°F
Ratio of raw to active waste -- 4:1
Original pH -- 5.6
Grams of CaO to raise pH -- 105
Air Gage Pressure -- 2.0 Psig.

Time of run 12 hours
Ml. of Na2S203 for blank 43.30
Amt. of waste treated 3760 g
Total oxygen required 546 g
G. of (NH4)2SO4 added 46.4
G. of NaH2PO4 added 30.7
Agitation rate (#1) 150 RPM.
G. of NH4NO3 added 31.4

TABLE III

EXPERIMENT 3

Variable - Air rate

Air Rate Investigated - 3.6 cu. ft/lb. COD/hr.

TIME	pH	AIR RATE	SOLIDS WEIGHTS			WT. OF COD SAMPLE	ML. OF Na2S2O3 REQUIRED
			After	Before	Tare		
2:15 A.M.	6.65	2.0	3.08	11.06	1.64	1.285	19.9
5:15 A.M.	7.25	2.0	3.54	13.12	1.65	1.365	17.6
8:15 A.M.	7.05	2.0	3.26	11.60	1.64	1.315	18.3
11:15 A.M.	6.85	2.0	3.72	14.33	1.54	1.15	21.45
2:15 P.M.	6.90	2.0	3.09	10.55	1.54	1.255	18.4
2:15 A.M.	6.65	2.0	3.66	13.38	1.51	1.28	17.6

Approximate Temp.	-- 130°F
Ratio of raw to active waste	-- 4:1
Original Ph	-- 5.7
Grams. of CaO to raise pH	-- 103
Air gage pressure	-- 20 psig.

Time of run	24 hours
Ml. of Na2S2O3 for blank	43.35
Amt. of waste to be treated	3475
Total oxygen required	548
G. of (NH4)2SO4 added	46.4
G. of NaH2PO4 added	30.7
Initial COD of waste	0.157
Agitation rate (#1)	150 RPM.
G. of NH4NO3 added	31.4

TABLE IV

EXPERIMENT 4

Variable - Air Rate

Air Rate Investigated - 6.5 cu. ft/lb. COD/Hr.

TIME	pH	AIR RATE	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S203 REQUIRED
6:30 P.M.	6.85	3.7	2.79	10.34	1.72	1.27	20.1
9:30 P.M.	7.20	3.7	2.60	8.58	1.69	1.485	13.45
12:30 A.M.	7.0	3.7	2.73	9.12	1.68	1.21	18.70
6:30 A.M.	6.6	3.7	2.915	9.920	1.68	1.165	17.80

Approximate Temp.	-- 130°F	Time of run	12 hours
Ratio of raw to active waste	-- 4:1	Ml. of Na2S203 for blank	43.30
Original pH	-- 5.6	Amt. of waste to be treated	3470 g
Grams.of CaO to raise Ph	-- 105	Total oxygen required	546 g
Air gage pressure	-- 3.8 psig.	G. of (NH4)2S04 added	46.4
		Initial COD of waste	0.1575
		Agitation rate (#1)	150 RPM.
		G. of NaH2P04 added	36.7
		G. of NH4N03 added	31.4

TABLE V

EXPERIMENT 5

Variable - Agitation

Agitation Speed - 315 R. P. M. (#4)

TIME	pH	AIR RATE	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2Sa03 REQUIRED
12:00P.M.	7.0	2.0	2.88	10.42	1.44	0.90	28.05
3:00 P.M.	6.25	2.0	2.575	8.230	1.43	1.175	21.25
9:00 P.M.	6.15	2.0	2.71	8.69	1.45	1.135	21.20
12:00 A.M.	5.95	2.0	2.69	9.15	1.40	1.095	22.65

Approximate Temp.	--	130° F	
Ratio of raw to active waste	-	4:1	
Original pH	--	5.7	
Grams of CaO to raise pH	--	100	
Air rate		3.6 cu.ft/lbCOD/hr.	
Air gage pressure		2.0 psig.	
Time of run			12 hours
Ml. of Na2S203 for blank			43.40
Amt. of waste to be treated			3750 g
Total oxygen required			548 g.
G. of (NH4)2S04 added			46.4
Initial COD of waste			0.146
G. of NaH2P04 added			30.7
G. of NH4N03 Added			31.4

TABLE VI

EXPERIMENT 6

Variable - Temperature

Temperature Investigated - 150°F

TIME	pH	TEMPERATURE ° F.	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF $Na_2S_2O_3$ REQUIRED
11:30 P.M.	7.2	150°	3.49	13.39	1.63	1.37	19.0
8:30 A.M.	6.6	150°	4.52	17.95	1.63	1.655	7.6
12:00 P.M.	6.5	150°	4.0	13.96	1.69	1.215	16.7

Ratio of raw to active waste -- 4:1
Original pH -- 5.6
Grams.of CaO to raise pH -- 105
Air rate -- 3.6 cu.ft/lb.BOD/hr.
Air gage pressure -- 2.0 psig.

Time of run	12 hours
ml.of $Na_2S_2O_3$ for blank	43.30
Amt. of waste to be treated	3590 g
Total oxygen required	545 g
G. of $(NH_4)_2SO_4$ added	46.4
Initial COD of waste	0.152
G. of NaH_2PO_4 added	30.7
Agitation rate (#4)	315 RPM.
G. of $(NH_4)NO_3$ added	31.4

TABLE VII

EXPERIMENT 7

Variable - Temperature

Temperature Investigated - 90° F.

TIME	pH	TEMPERATURE o F	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S203 REQUIRED
6:00 P.M.	6.9	90°	3.42	13.36	1.68	1.26	20.85
12:00 A.M.	7.0	90°	3.75	14.32	1.67	1.20	18.65
8:00 A.M.	6.9	90°	4.00	15.20	1.60	1.21	18.35

Ratio of raw to active waste -- 4:1
Original Ph -- 5.5
Grams of CaO to raise pH -- 115
Air Rate -- 3.6 cu. ft/lb. COD/hr.
Air gage pressure -- 2.0 psig.

Time of run	12 hours
Ml. of Na2S203 for blank	43.25
Amt. of waste to be treated	3580 g
Total oxygen required	548 g
G. of (NH4)2S04 added	46.4
G. of NaH2P04 added	30.7
Initial COD of waste	0.153
Agitation rate (#4)	315 RPM.
G. of NH4N03 added	31.4

TABLE VIII

EXPERIMENT 8

Variable - Temperature

Temperature Investigated - 112° F.

TIME	PH	TEMPERATURE o F.	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S2O3 REQUIRED
12:00 P.M.	6.4	112°	2.855	10.670	1.430	1.185	21.65
3:00 P.M.	7.0	112°	2.615	9.540	1.435	1.550	14.0
6:00 P.M.	7.2	112°	2.74	8.68	1.43	1.145	20.90
9:00 P.M.	7.1	112°	2.490	8.210	1.445	1.185	21.10
12:00 A.M.	7.0	112°	2.500	10.455	1.460	1.30	24.70

Ratio of raw to active waste -- 4:1
Original pH -- 5.8
Grams of CaO to raise pH -- 95
Air rate - 3.6 cu. ft/lb.COD/hr.
Air gage pressure -- 2.0 psig.

Time of run 12 hours
Ml. of Na2S2O3 for blank 43.40
Amt. of waste to be treated 3480 g
Total oxygen required 546 g
G. of $(NH_4)_2SO_4$ added 46.4
G. of NaH_2PO_4 added 30.7
Agitation rate (#4) 315 RPM.
G. of NH_4NO_3 added 31.4

TABLE IX

EXPERIMENT 9

Variable - Ratio of Active Seed Waste to Raw Coffee Waste

Ratio Used - 2:1

TIME	pH	TEMPERATURE o F.	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S2O3 REQUIRED
1:00 A.M.	7.15	130°	2.98	11.705	1.44	1.205	17.3
7:00 A.M.	7.00	130°	3.61	12.84	1.70	1.095	15.95
10:00 A.M.	7.00	130°	4.33	15.59	1.71	1.305	10.70
1:00 P.M.	6.9	130°	4.32	15.51	1.71	1.295	9.90

Original Ph - 5.7
Grams of Cao to raise Ph - 95
Air rate - 2.52 cu. ft/lb. COD/Hr.
Air gage pressure - 2.0 psig.

Time of run	12 hours
Ml. of Na2S2O3 for blank	43.40
Amt. of waste to be treated	3470 g.
Total oxygen required	642 g.
G. of (NH4)2SO4 added	21.8
G. of NaH2PO4 added	14.5
Agitation rate (#4)	315 RPM.
G. of NH4NO3 Added	14.8
G. of raw coffee waste added	200 g.

TABLE X

EXPERIMENT 10

Variable - Ratio of Active Seed Waste to Raw Coffee Waste

Ratio Used - 1:1

TIME	pH	TEMPERATURE o F.	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S203 REQUIRED
3:45 P.M.	7.0	130°	4.72	18.04	1.71	0.936	18.05
7:30 P.M.	6.85	130°	4.31	15.14	1.71	1.130	12.10
10:30 P.M.	6.60	130°	3.69	11.56	1.70	1.000	15.10
4:00 A.M.	6.65	130°	3.99	12.60	1.69	0.885	16.60

Original Ph - 5.3
Grams of CaO to raise pH - 120
Air rate - 2.42 cu. ft/lb COD/hr.
Air gage pressure - 2.0 psig.

Time of run	12 hours
Ml. of Na2S203 for blank	43.25
Amt. of waste to be treated	3510 g.
Total oxygen required	800 g.
G. of (NH4)2S04 added	32.8
G. of NaH2P04 added	21.6
Agitation rate (#4)	315 RPM.
G. of NH4N03 added	220
G. of Raw Coffee Waste added	300

TABLE XI

EXPERIMENT 11

Variable - Ratio of Active Seed Waste to Raw Coffee Waste

Ratio Used - 1:2

TIME	pH	TEMPERATURE o F.	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S203 REQUIRED
6:30 A.M.	7.05	130°	3.66	12.15	1.66	0.929	15.7
9:30 A.M.	7.0	130°	4.26	15.07	1.67	1.115	12.3
1:00 P.M.	6.7	130°	4.60	16.19	1.67	1.155	9.95
4:00 P.M.	6.7	130°	3.73	11.74	1.68	0.970	15.2
6:30 P.M.	6.1	130°	3.65	11.83	1.67	1.250	6.1

Original Ph -- 5.4
Grams of CaO to raise Ph -- 115
Air rate - 2.04 cu. ft/lb COD/hr.
Air gage pressure -- 2.0 psig.

Time of run	12 hours
Ml. of Na2S203 for blank	43.30
Amt. of waste to be treated	3540 g.
Total oxygen required	905 g.
G. of (NH4)2SO4 added	43.6
G. of NaH2PO4 added	29.0
Agitation rate (#4)	315 RPM.
G. of NH4NO3 added	29.6
G. of raw coffee waste added	400 g.

TABLE XII

EXPERIMENT 12

Variable - Waste Conditioning
Coffee Waste Autoclaved

TIME	pH	TEMPERATURE o F.	SOLIDS WEIGHTS After	Before	Tare	WT. OF COD SAMPLE	ML. OF Na2S2O3 REQUIRED
9:15 P.M.	7.25	130°	3.02	11.67	1.44	0.975	20.55
12:15 A.M.	7.20	130°	3.32	13.32	1.44	0.970	22.4
6:15 A.M.	6.8	130°	3.29	11.93	1.44	0.940	20.0
9:30 A.M.	6.75	130°	3.320	11.670	1.435	0.775	23.7

Original pH -- 6.1
Grams of CaO to raise pH -- 80
Air rate - 2.8 cu. ft/lb. COD/hr.
Air gage pressure -- 2.0 psig.

Time of run	12 hours
Ml. of Na2S2O3 for blank	43.40
Amt. of Waste to be treated	3455 g
Total oxygen required	695 g
G. of (NH4)2SO4 added	32.8
G. of NaH2PO4 added	21.6
Agitation rate (#4)	315 RPM.
G. of NH4NO3 added	22.0
G. of raw waste added	300

VI. TABLES AND GRAPHS (continued)

B. Calculation Tables and Graphs

TABLE XIII

Variable - Air Rate

Summary of Calculations
Air Rate Varied - 0.9 to 6.5 cu. ft/ lb. COD/Hr.

EXPERIMENT NO.	AIR RATE	ELAPSED TIME (HRS)	COD	PERCENT SOLIDS	CORRECTED COD	PERCENT COD LOSS OR GAIN
1	0.9	0	0.1605	17.2	0.1605	0
		3	0.1510	16.7	0.1550	-3.1
		7 1/4	0.1695	16.7	0.1702	6.1
		9	0.1565	16.4	0.163	1.5
		12	0.1510	16.7	0.1608	0.1
2	1.8	0	0.1455	13.8	0.1455	0
		3	0.1960	15.0	0.180	12.4
		6	0.155	14.5	0.148	1.7
		12	0.163	14.5	0.155	6.9
3	3.6	0	0.157	15.3	0.157	0
		3	0.168	16.5	0.156	-0.6
		6	0.164	16.3	0.154	-1.3
		9	0.164	17.1	0.147	-6.3
		12	0.171	17.2	0.152	-3.2
		24	0.180	18.2	0.151	-3.8
4	6.5	0	0.1575	12.4	0.1575	0
		3	0.172	13.2	0.161	2.1
		6	0.175	14.2	0.153	-2.9
		12	0.188	15.0	0.155	-1.7

FIGURE 5
AIR RATE DETERMINATION
VARIED - 0.9 to 6.5 Cu.Ft./lb COD/Hr.

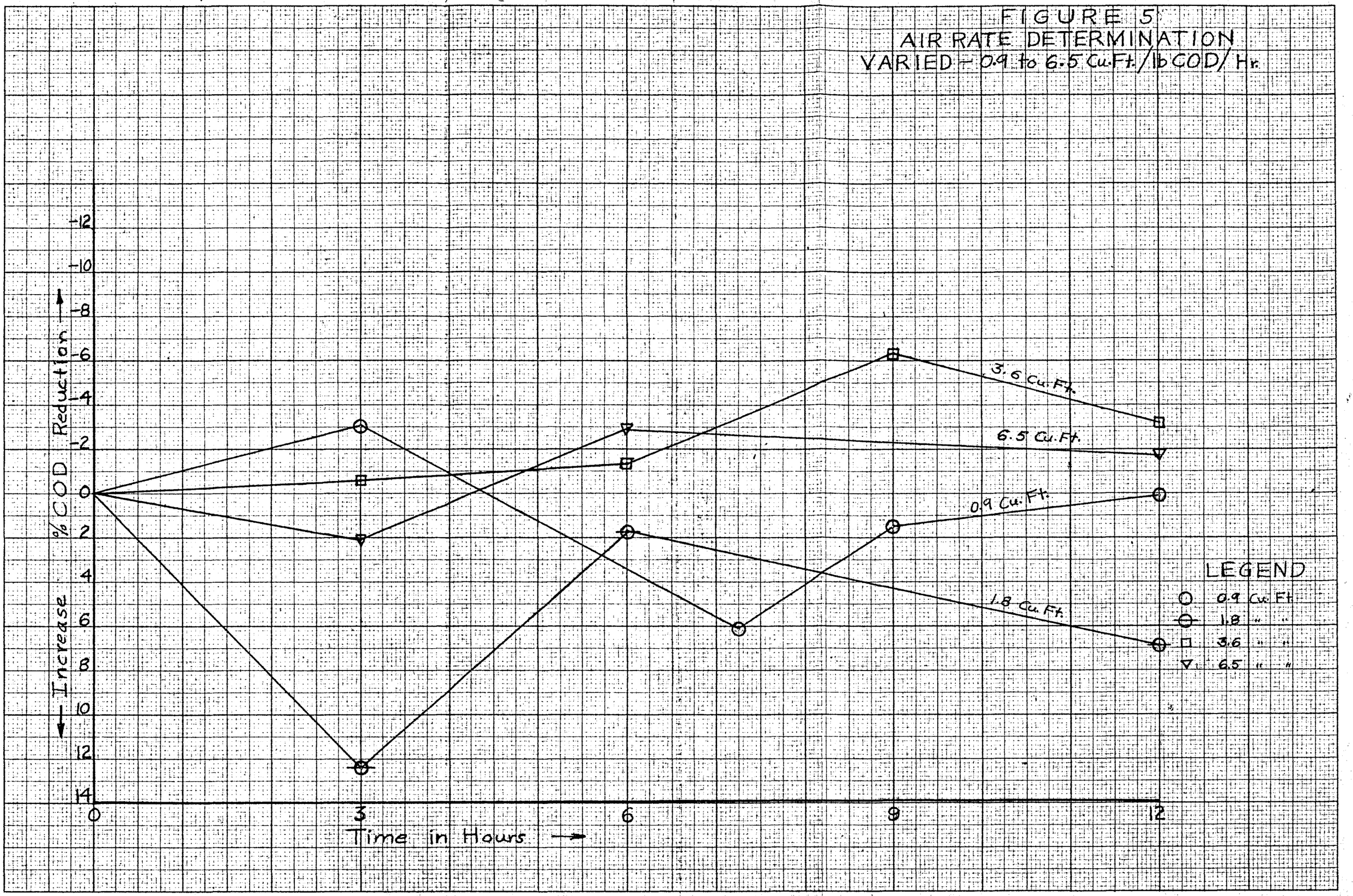


TABLE XIV

Variable - Agitation

Summary of Calculations

Agitation Speed Varied - 150 to 315 RPM.

EXPERIMENT NO.	AGITATION SPEED	ELAPSED TIME (HRS)	COD	PERCENT SOLIDS	CORRECTED COD	PERCENT COD LOSS OR GAIN
# 3	150	0	0.157	15.3	0.157	0
		3	0.168	16.5	0.156	-0.6
		6	0.164	16.3	0.154	-1.3
		9	0.164	17.1	0.147	-6.3
		12	0.171	17.2	0.152	-3.2
5	315	0	0.146	16.1	0.146	0
		3	0.162	16.8	0.155	6.1
		9	0.168	17.4	0.155	6.1
		12	0.164	16.7	0.158	8.1

FIGURE 6
AGITATATION DETERMINATION
VARIED - 150 to 315 RPM

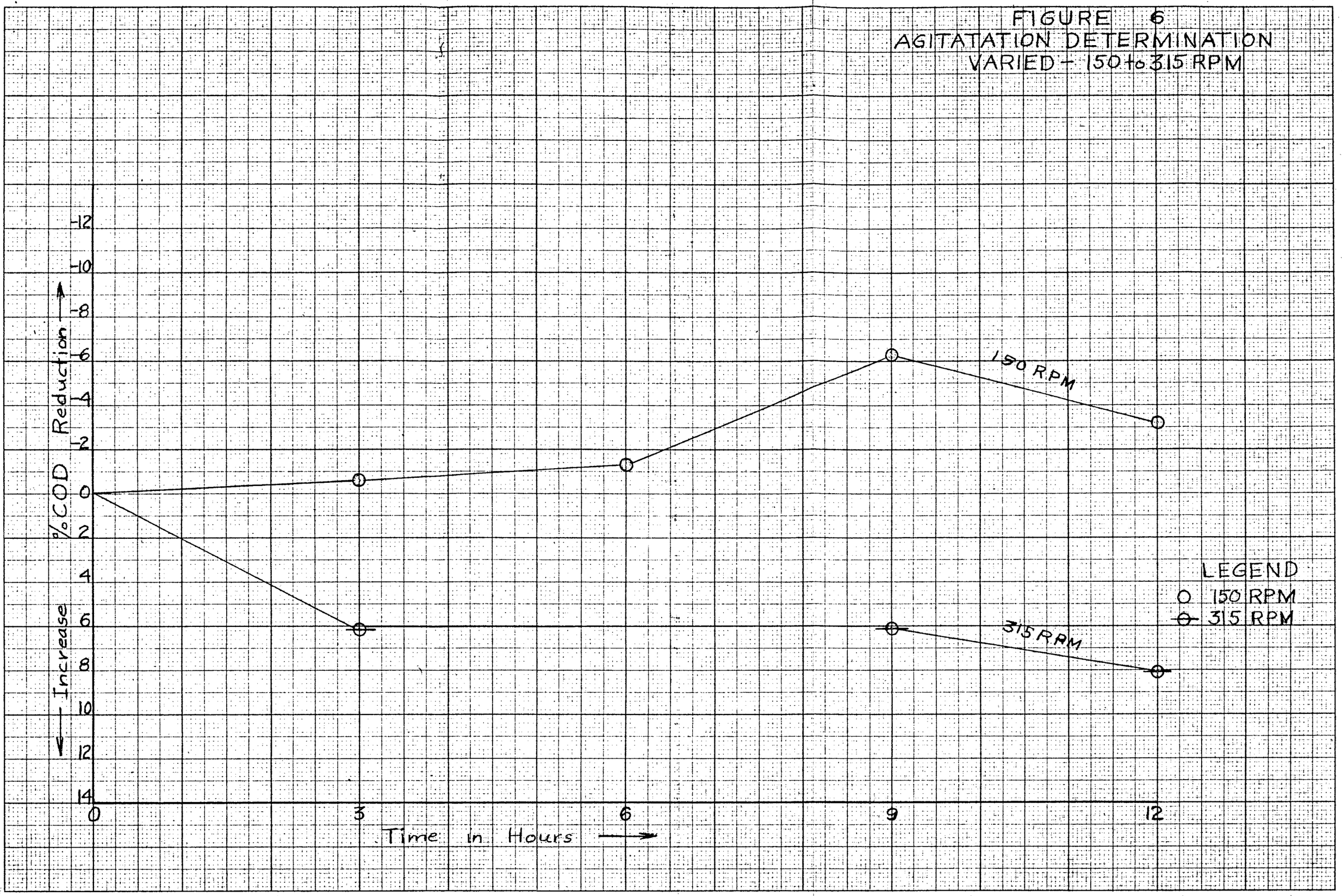


TABLE XV

Variable - Temperature

Summary of Calculations
Temperature Varied - 90° to 150° F.

EXPERIMENT NO.	TEMPERATURE ° F.	ELAPSED TIME (HRS)	COD	PERCENT SOLIDS	CORRECTED COD	PERCENT COD LOSS OR GAIN
7	90	0	0.153	14.9	0.153	0
		6	0.177	16.45	0.160	4.5
		12	0.178	17.7	0.150	-2.1
8	112	0	0.157	15.4	0.157	0
		3	0.162	14.6	0.173	11.1
		6	0.169	18.1	0.144	-8.0
		9	0.162	15.6	0.159	1.2
		12	0.123	11.4	0.166	5.7
5	130	0	0.146	16.1	0.146	0
		3	0.162	16.8	0.155	6.1
		9	0.168	17.4	0.155	6.1
		12	0.164	16.7	0.158	8.1
6	150	0	0.152	15.8	0.152	0
		9	0.194	17.7	0.173	11.38
		12	0.188	18.0	0.165	8.6

FIGURE 7
TEMPERATURE DETERMINATION
VARIED – 90° to 150°F

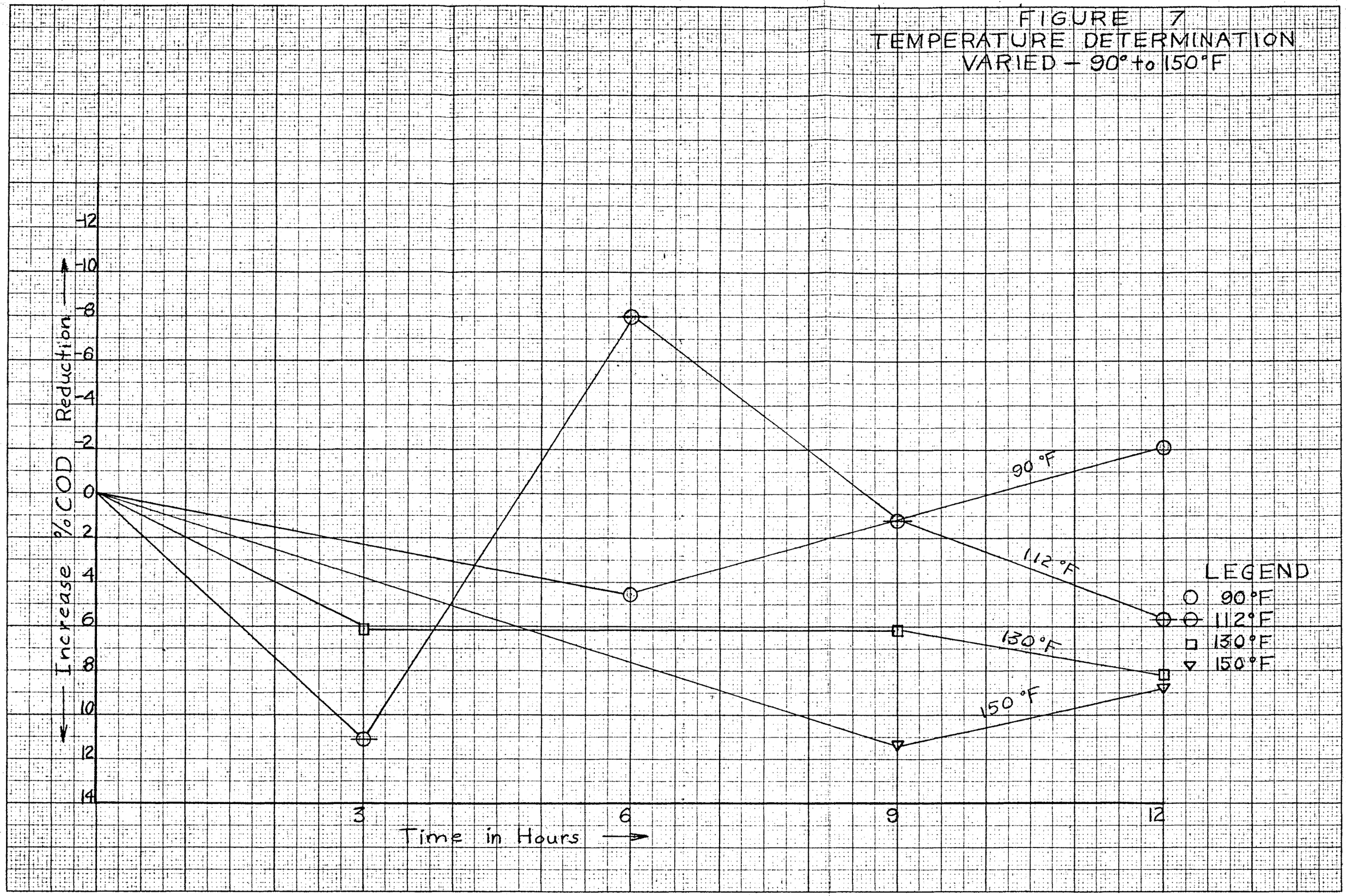


TABLE XVI

Variable - Ratio of Active Seed Waste to Raw Coffee Waste

Summary of Calculations
Ratio Varied - 1:4 to 2:1

EXPERIMENT NO.	RATIO USED	ELAPSED TIME (HRS)	COD	PERCENT SOLIDS	CORRECTED COD	PERCENT COD LOSS OR GAIN
5	1:4	0	0.146	16.1	0.146	0
		3	0.162	16.8	0.155	6.1
		9	0.168	17.4	0.155	6.1
		12	0.164	16.7	0.158	8.1
11	1:2	0	0.256	19.1	0.256	0
		3	0.240	19.3	0.237	-7.4
		6.5	0.249	20.2	0.235	-8.1
		9.5	0.249	20.3	0.234	-8.3
		12	0.256	19.5	0.251	-1.9
10	1:1	0	0.228	18.4	0.228	0
		4	0.238	19.3	0.227	-0.8
		7	0.231	20.1	0.220	-3.8
		12	0.235	21.4	0.221	-3.3
9	2:1	0	0.186	14.9	0.186	0
		6	0.215	17.1	0.187	0.5
		9	0.215	18.9	0.170	-8.7
		12	0.222	18.9	0.173	-7.0

FIGURE 8
RATIO of ACTIVE to RAW WASTE
DETERMINATION
VARIED — 1:4 to 2:1

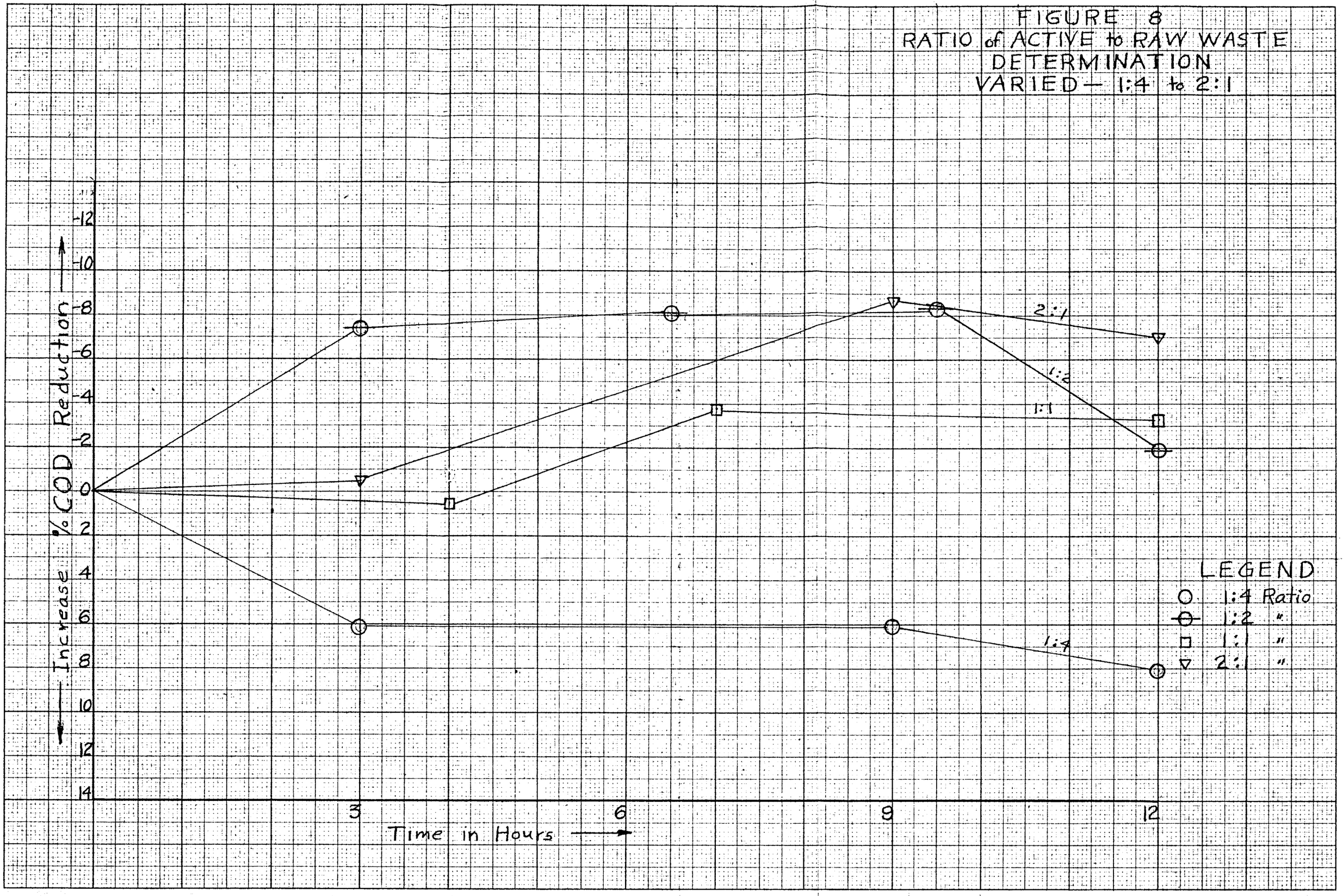


TABLE XVII

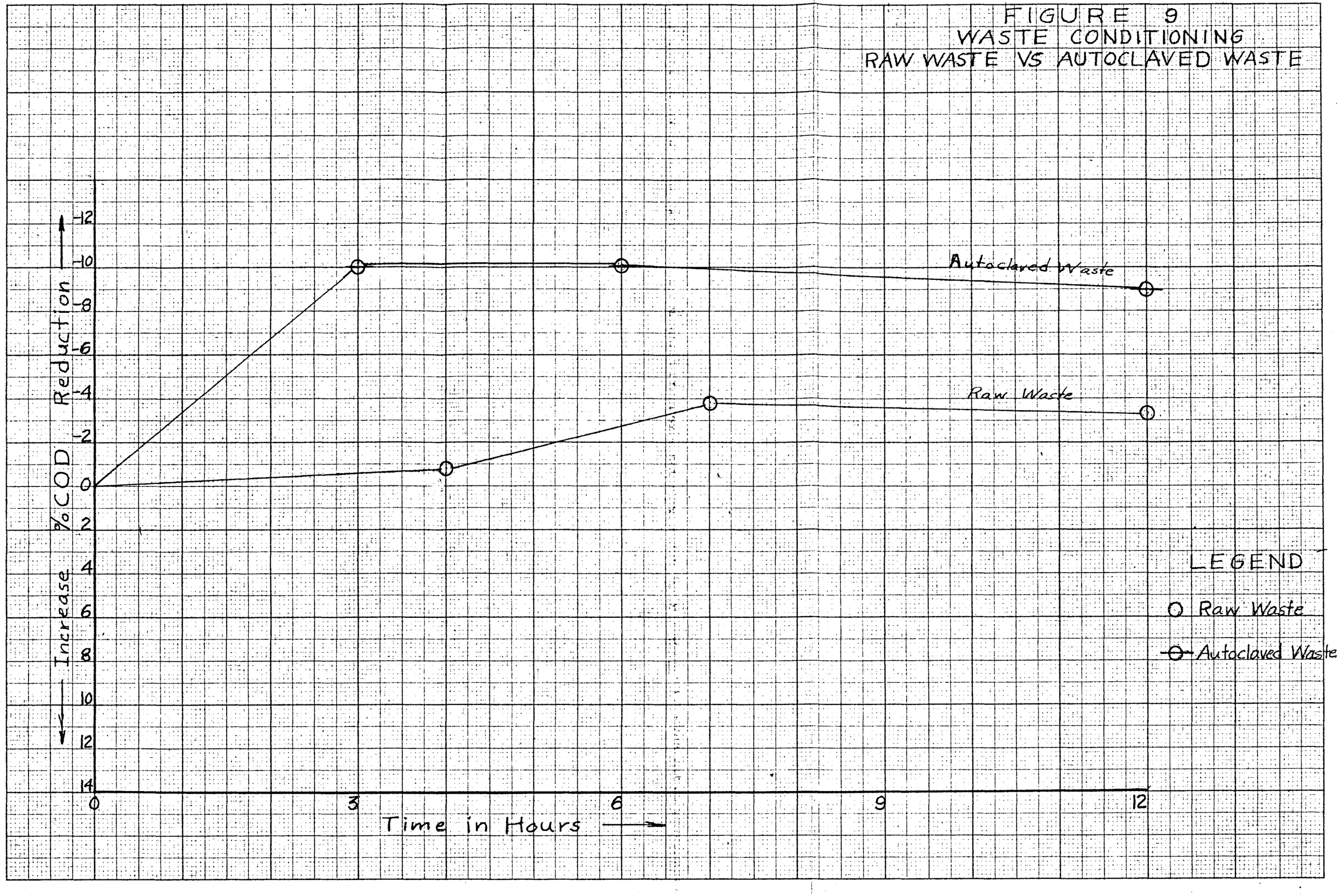
Variable - Waste Conditioning

Summary of Calculations

Raw Coffee Waste Versus Autoclaved Coffee Waste

EXPERIMENT NO.	WASTE PRETREAT-MENT	ELAPSED TIME(HRS)	COD	PERCENT SOLIDS	CORRECTED COD	PERCENT COD LOSS OR GAIN
10	none	0	0.228	18.4	0.228	0
		4	0.238	19.3	0.227	-0.8
		7	0.231	20.1	0.220	-3.8
		12	0.235	21.4	0.221	-3.3
12	autoclaved	0	0.201	15.4	0.201	0
		3	0.185	15.9	0.180	-10.0
		6	0.206	17.7	0.179	-10.1
		12	0.218	18.4	0.182	-9.0

FIGURE 9
WASTE CONDITIONING
RAW WASTE VS AUTOCLAVED WASTE



VII. RESULTS AND DISCUSSION

Table XVIII summarizes the variables investigated including the ranges covered and the optimum values obtained.

A. General

Prior to composting, the coffee waste slurry contained some hard particles and was brown in color. After digestion, the compost produced was dark in appearance and very fine in particle size. The odor of the final material was of a moldy, slightly fermented nature. Moist compost dried to a fine and fluffy dark brown powder. Wiley (85) gives a similar description of his composted garbage and refuse.

The actual value of the final product for use as an organic fertilizer was not determined because it involved observing the effect of the compost on the growth of various crops and this observation was beyond the scope of this study. However, it is taken for granted (45) that a satisfactory compost is one that has been decomposed to the extent where it has suffered a 50 to 60 percent loss in weight. Since loss in weight is directly proportional to reduction in chemical oxygen demand, a COD reduction of the same magnitude, namely 50 to 60 percent, would indicate adequate decomposition had taken place. In this investigation concerning composting coffee waste, an acceptable product was not obtained as the estimated maximum COD reduction of the residual material was only 20 to 25 percent.

On the average, the pH of all the experiments increased during the first three hours and then steadily decreased.

TABLE XVIII

Summary of Results

VARIABLE	RANGE INVESTIGATED	OPTIMUM VALUE	WILEY'S (82) OPTIMUM FIGURES
Air Rate	0.9 to 6.5 cu.ft./lb COD/hr.	3.6 cu.ft./lb COD/hr.	1.0 cu. ft./lb COD/hr.
Agitation	150 to 315 RPM	315 RPM.	No Optimum
Temperature	90° to 150° F.	130° F.	—
Ratio of Active To Raw Waste	1:4 to 2:1	2:1	—
Waste Autoclaving	no treatment vs. autoclaving	autoclaving	—

The pH of each run was always between the optimum range of 6.0 to 8.0. Wiley (85) reports that the pH of his runs start at 5.5 to 6.0 and drops to 4.5 or 5.0 in one day. It then increases to 8.0 to 8.5 at the top temperature of the run and at the end falls to 7.5 to 8.0. Wiley (85) did not take advantage of the literature information concerning optimum pH range (32).

Under optimum conditions, disregarding autoclaving waste, maximum reduction in chemical oxygen demand was 7.0 percent in a twelve hour period. Prior to undertaking this study, it was anticipated that a 40% reduction would be attained in the same period of time based on activated sludge studies (36) (58) (20). However, literature investigation after this study was completed revealed that these other determinations indicating high reductions (70 - 90%) of organic matter, were made on the clarified effluent and not on a mixture of the sludge and effluent. The activated sludge absorbed the soluble and finely dispersed organic waste but oxidized very little of it.

For a twenty-four hour period, the maximum rate is assumed to be 8.5 percent since in one run an additional twelve hours did effect an increase of the same magnitude. It is postulated that if the experiment was continued, decomposition of the waste would stop at the end of 4 to 6 days with a final reduction in COD of 20-25% which means the organic content is reduced by one-fourth. In his investigations, Wiley (85) states that composting of domestic garbage is over in 6 to 9 days with an average decomposition of 30%. The difference

in composting time is due to the fact that in Wiley's experiments, it takes three days to reach the optimum operating temperature of this study. Domestic garbage is not as complex in organics as coffee waste and would therefore be decomposed to a greater degree. Also, it is possible that the coffee oil and fats coat the bacteria thereby preventing micro-organisms from attacking the waste since a similar condition is observed in domestic garbage from fats and oils as reported by Gurnham (27). In addition, the concentration of toxic materials, including formic acid and hydroquinone, may be greater in coffee waste than in domestic garbage. A volatile solids determination in conjunction with the COD value, might have indicated that the rate of coffee waste decomposition was greater than reported.

Porges et al (58) (36) and Eckenfelder and O'Connor (20) reported that only 30% to 37% of the organic sludge is oxidized in the aerobic bioxidation process indicating that this process limits the decomposition of organic waste after a certain amount has been oxidized. These investigators and others (44) postulate it is possible that during the decomposition of the complex organic materials, stable breakdown products are produced which resist and prevent further catabolism to the ultimate end products of cell tissue, water and carbon dioxide. A BOD determination would not detect this semi-oxidized matter, but a COD or volatile solids examination would.

B. Specific

1. Air rate. The air rate was varied from 0.9 to 6.5 cubic feet per pound of COD per hour with the optimum value determined at 3.6 which produced a reduction in COD of 3.2%. Wiley reports an optimum air rate of 10 to 30 cubic feet per day per pound of volatile solids. This would average out to 0.5 to 1.0 cubic feet per pound of COD per hour. However, at high air rates, Wiley did not maintain sufficient water in the digestor to support the bioxidation of waste and hence little decomposition took place. Therefore, high air rates may have affected increased decomposition of the organic waste had adequate moisture conditions existed.

In this study, low air rates showed an increase in COD rather than a decrease. This is explained by the fact that there is 25% more organic material than was available to determination by the COD method. As the bacteria attacked the waste, micro-organisms reduced the complex organic substances to simple ones which were detected by the COD method indicating an increase in oxygen demand over initial results. This breakdown at low air rates proceeded at a greater rate than the oxidation of the organic waste due to less oxygen available. This is supported to some extent by Porges (36) and Eckenfelder (20) who state that the rate of food assimulation and storage is greater than the oxidation of the organic matter. As the air rate is increased, the oxidation

rate increases to a level where the optimum value is obtained.

2. Agitation. The agitation rate was varied from 150 to 315 RPM. with the higher speed being the optimum one. This speed gives increases in COD rather than reductions which is the result of greater activity of the micro-organisms in breaking down or assimilating the complex organic matter. Porges et al (58) reports that in a vigorously agitated aerator the storage and assimilation was faster initially than the oxidation rate.

In his paper, Wiley informs his reader that no effect is observed with the agitation ranges investigated. However, he states, this is probably due to the low ranges selected and that high agitation rates would be beneficial.

3. Temperature. In the range investigated (90° to 150°F.), increasing temperature indicated an increase in COD determinations at optimum air rate and agitation. The biological activity in the direction of organic complexity breakdown was attained at increasing temperatures with the optimum value between 130° to 150° F. Porges et al (58) and Gellman and Heukelekian (25) reported that high temperatures increase greatly the storage and assimilation tendencies of the aerobic micro-organisms.

Erratic high and low COD values were obtained on the 112°F. run. These figures might have been due to poor sampling technique, inadequate agitation or localized activity.

The author can give no concrete reason for the deviation.

4. Ratio active seed waste to raw coffee waste. This ratio was varied from proportions of 1:4 to a final value of 2:1. The latter experiment using a ratio of two parts active to one raw was considered to be an optimum run. The large number of bacteria present created a condition where the oxidation of the waste was greater than the breakdown of the organic substances. The maximum COD reduction was 7.0% under optimum conditions. Heukelekian (31) reports the same ratio has been used successfully in the aeration of soluble organic waste with nonflocculent growths.

5. Conditioning of waste by autoclaving. Conditioned waste was composted according to optimum conditions of air rate, agitation and temperature. The ratio of active to conditioned waste was 1:1. In comparing the results with a similar run using untreated waste, it was observed that the percent COD reduction increased from 3.3% to 9%. Autoclaving of the coffee waste succeeded in breaking down the complex matter and consequently, greatly increasing the decomposition rate since the waste was more readily susceptable to oxidation.

VII. CONCLUSIONS

1. All of the variables evaluated have an effect on the composting of coffee waste. The optimum values for each are listed in table XVIII.

2. Autoclaving of coffee waste prior to composting,

greatly increases the decomposition rate.

3. The low oxidation rates and low estimated final decomposition of 20 to 25% are due to:

a. Primarily the aerobic process which limits the amount of oxidation by forming stable compounds.

b. Natural complexity of coffee waste.

c. Possible effect of coffee oil and fats coating bacteria and preventing them from attacking the waste (27).

d. Toxic materials.

4. The aerobic process is judged an unsuitable method for producing a satisfactory compost from coffee waste based on the high concentration of organic matter left undecomposed according to percent COD reduction.

IX. RECOMMENDATIONS

1. It is recommended that investigations of coffee waste decomposition be conducted for a period of four days, or longer if necessary, under the optimum conditions of this study to ascertain if the estimated final reduction is correct.

2. It is recommended that a correltaion of the COD and volatile solids of coffee waste be determined so as to obtain a more accurate idea of the decomposition rate.

3. It is recommended that the results, discussion and conclusions of this paper be published to support the supposition that the aerobic process is not suitable for decomposition of concentrated organic wastes.

4. It is recommended that the anaerobic method be evaluated in the decomposition of coffee waste with respect to the variables of agitation, temperature, micro-organism concentration and waste conditioning.

X. APPENDIX

A. 1. Chemical Oxygen Demand Procedure

Reference: "APPLICATION OF A RAPID CHEMICAL DEMAND TEST FOR DETERMINING ORGANIC POLLUTIONS," by B. Pepinsky, Nandor Porges and Sam R. Hoover. Proceedings of the Sixth Industrial Waste Conference, February 21 to 23, 1951, Series No. 76, Purdue University.

Reagents

1.) Dichromate oxidizing agent is prepared by dissolving 20 to 25 g. of potassium dichromate in a mixture of 500 ml. each of concentrated H_2SO_4 and 85% ortho H_3PO_4.

2.) Potassium iodide solution is made by dissolving 55.3 g. of KI in 200 ml. of distilled water.

3.) Sodium thiosulfate solution 0.1 N.(24.82 of $Na_2S_2O_3$ $5H_2O$ per liter.

4.) Starch solution--1% good grade of soluble starch which gives a true blue color with iodine.

Procedure

1.) Pipette exactly 50 ml. of the dichromate oxidizing solution in a 500 ml. Phillips beaker.

2.) Weigh out on a micro-balance (3 places) 0.9 to 2.0 grams of waste slurry and place in Phillip's Beaker tilted on an angle. Add 5 cc. of distilled H_2O.

3.) Place beaker on hot plate which has been preheated to the required temperature and suspend thermometer in solution.

4.) Heat with frequent swirling so that the temperature of 165°C is reached in six minutes.

5.) Remove to water bath and cool to approximately room temperature.

6.) Transfer contents of beaker to 500 ml. volumetric flask being careful to wash beaker throughly with distilled water. Bring to mark with distilled water and mix by vigorous shaking.

7.) A 50 ml. aliquot portion is transferred back to a clean Phillips Beaker.

8.) Add 200 cc. of distilled water to beaker.

9.) Add 15 ml. of the iodide solution and titrate with 0.1N sodium thiosulfate, adding the starch near the end point. The color changes from blue to green.

10.) A blank determination of aliquot portion of oxidizing solution in which 5 ml. of distilled water is used, is run through the same procedure.

11.) The total chemical oxygen demand expressed as parts per million or as in the case of the following formulation as . of oxygen per gram of slurry waste.

$$COD = \frac{(10)\ (\text{ml } Na_2S_2O_3 \text{ in blank} - \text{ml } Na_2S_2O_3 \text{ in sample})(\text{Norm.})(8)}{(1000)\ (\text{Wt. of sample})}$$

2. Coffee Component Determination

a. Nitrogen Determination

Procedure: Weigh from 0.9 to 1.10 grams of sample into a clean, dry glass vial. Prepare an 800 ml. kjeldahl flask so that it contains 15 frams of anhydrous sodium sulfate, a few crystals of copper sulfate, 35 ml. of concentrated sulfuric acid, and 2 or 3 Hengar selenized granules. Tilt the flask and slide the vial down the neck of the flask into

the digestion mixture.

Digest the samples, slowly at first, strongly at the end, until a clear green to blue-green solution is obtained. Allow the flasks to cool, dilute with distilled water to one-third the volume of the flask and set up in a vertical position for distillation. To the receiving flasks, add a 50 ml. aliquot of 0.1N H_2SO_4 and a few drops of methyl red and arrange the delivery tube so that it barely touches the surface of the liquid.

To the kjedahl flasks, very quickly add in succession, a few zinc turnings and 100 ml of a 30-50% solution of NaOH, and immediately close the flasks. Before beginning the distillation, be sure that the material in the flask looks either blue or a muddy violet.

Distill over about 100 ml and then wash the material in the condenser down into the receiver. Titrate the unreacted H_2SO_4 with 0.1N KOH to a yellow end point.

Calculation: $\%N = \frac{100\ (\text{ml } H_2SO_4 - \text{ml } 0.1N\ KOH)(.0014)}{\text{Weight of Sample}}$

$$\% \text{ Protein} = (\% \text{ total N})\ (6.25)$$

b. Phosphorus Determination

Procedure: Weigh out 1.0 to 1.4 g. of sample and place in a 500 ml wide-mouth flask. Add several glass beads, 10 ml. of conc. sulfuric acid and 20 ml. of conc. nitric acid.

Boil mixture on a strong, short-coned flame until charring begins. Successive 5 ml. additions of conc. nitric acid are made to the fuming solution until the liquid appears straw-yellow in color. At this point, two successive additions

of 5 ml. of superoxal are added turning fuming concentration to water-white color.

Cool and filter into 400 ml. beakers. Neutralize with conc. NH_4OH (using litmus paper) and re-acidified with 5 ml. of conc. HNO_3. Adjusted solution is heated to 80°- 90°C and 50 ml of ammonium molybdate reagent are added with constant stirring. Beaker is then placed on hot plate for half an hour to coagulate precipitate.

The precipitate is filtered hot on a Moore-Shimer funnel (Cenco #15196, padded with 1/4" layer of filter pulp). It is then washed with hot distilled water until the filtrate no longer turns blue litmus paper red.

The funnel is inverted in the neck of a wide-mouth 500 ml. Erlenmeyer flask and the pad and ppt. are pushed out of the funnel. The funnel is throughly washed into the flask using additional hot water.

The phospho-molybdate is dissolved with 35 ml. of N/2 sodium hydroxide and a 15 ml. excess is added. When all of the yellow precipitate has been dissolved by vigorous shaking, the excess alkali is back titrated with N/4 nitric acid using Phenolphthalein as the indicator.

$$\% P_2O_5 = \frac{(\text{ml. N/2 NaOH} - \frac{\text{ml. N/4 } HNO_3}{2})(0.1544)}{\text{Weight of sample}}$$

c. Ash Determination

Grind sample to pass through 30-mesh sieve. Heat sample of appropriate weight for product being examined (usually 5 - 10 g.) in 50-100 ml. platinum dish at 100° C. until all

water is expelled; add a few drops of pure olive oil and heat slowly over flame until swelling ceases. Place dish in muffle furnace at 525°C. and leave (1-2 hrs.) until white ash is obtained. Moisten ash with H_2O, dry on steam bath and then on hot plate, and re-ash in muffle furnace at 525°C. to constant weight.

$$\% \text{ Ash} = \frac{\text{Weight of ash}}{\text{Weight of sample}}$$

d. Oils, Fats and Waxes

Dry 2 grams of sample at 100°C. until all the water is expelled. Treat with petroleum ether (bip. 35-50°C.) for one hour under agitation. Filter on buchner funnel and wash cake well with ether. Dry cake on steam bath to constant weight.

$$\% \text{ Oils, Fats \& Waxes} = \frac{\text{Weight of sample - weight of cake}}{\text{Weight of sample}}$$

e. Crude Fibre

% Crude fibre = 100 - (% ash plus % protein plus % oils, fats and waxes)

Reference: "OFFICIAL METHODS OF ANALYSIS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS", 7th edition, published by the Association of Official Agricultural Chemists, Washington 4, D. C. (1950).

B. Equipment Specifications

1. Digestor (figure 1)

Material of construction - pyrex glass.

Inside diameter - 3 1/8"

Outside diameter - 4 3/4"

Length - 36"

Wall thickness - 1/8"

Resistance Wire - #20 B&S gauge, asbestos covered 0.635 Ohms/in., wrappings spaced 1/2" apart on digestor.

2. Bottom fixture (figure 2)

Rubber stopper - No. 14

Thermometer - 0-100°C., No. 10/30 tapered joint; 10-inch stem, Fisher Cat. No. 15-002

Air sparger - pyrex fritted glass, coarse size, 12 mm. o.d., Fisher Cat. No. 11-138

Sample tube - 11 mm. Pyrex test tube with bottom cut off, 1/8" glass rod with cork stopper on end.

3. Agitator and stirrer

Motor type - variable speed direct worm drive

Speed - 200 to 1000 RPM.

Rated voltage - 115 a.c. or d. c.

Fisher Cat. No. 14-499

Stirrer - 1/8" stainless steel rod fitted with wire paddles spaced one inch apart, rod is 36" long.

4. Air Compressor

Fisher Cat. No. 1-093-5

Description - pressure and vacuum, motor driver with gauges and handle.

Actual free air delivery - 1.3 cu. ft. at 10 psig.

Continuous service - 15 psig. maximum.

5. Reserve tank

Material of construction - steel

Capacity - 15 gallons

Maximum operating pressure - 15 psig.

6. Pressure regulator

Fisher Cat. No. 11-163

Capacity - 20 cu. ft.

Operating range - 5 to 25 lbs. psi.

7. Flowmeter

Fisher Cat. No. 11-163

Operating capacity - 0 to 3 1/2 liters per minute

Accuracy - within 1 1/2% of scale reading

8. Water bath

Make - American Instrument Company

Model No. 4-56

Voltage - 110

Wattage - 1700

Temperature control - metastatic mercury thermoregulator.

Capacity - 5 gallons

9. Centrifugal pump

Fisher Cat. No. 13-874-92

Type - midget circulating Model A

Capacity - 4 to 6 gal./min. at 10-12 psig.

Horsepower - 1/100 h.p.

Voltage - 115 a.c. or d.c.

Resistor - sliding contact. Fisher Cat. No. 9-528D, 1.1 amps, 360 ohms.

10. Ph meter

Fisher Cat. No. 11-505-301 Make - Beckman Model N-1, portable

11. Analytical scale

Fisher Cat. No. 2-022

Description - heavy duty, triple beam balance

Range - 0.01 g. to 100 g.

12. Hot plate

Fisher Cat. No. 11-468-5

Power consumption - 235 watts, 3 heat.

13 Comminuting Machine

Make - Fitzpatrick

Speed - 1800 RPM.

Screen - triple zero perforated screen.

C. Glossary

Absorption - Process by which food or raw material pass into the bacteria cells.

Acclimatization - Process by which the raw waste becomes conditioned to the organism environment.

Activated Sludge - Inert suspended sewage combined with bacteria active in the oxidative process.

Active Waste - Inert suspended raw waste combined with bacteria active in the oxidative processes.

Adaptation - Modification to perform a specialized activity.

Aerobic Oxidation - Process which uses free oxygen for normal activity.

Amylose - A starch splitting enzyme which hydrolyzed complex sugars to glucose.

Anabolism - Process of changing food into cell tissues where heat is absorbed.

Anaerobic Oxidation - Process not requiring oxygen for normal activity.

Assimulation - Process by which some digested food is converted to cell matter called protoplasm.

Autoclaving - Process of inducing chemical reaction to occur under high pressure.

Bacteria - A group of microscopic, one-celled fungus plants.

Bacteria, Zoogloea - Irregular masses of bacteria held together by a common gelatinous secretion.

Bacterialogical Conversion - Process of conversion through action of bacteria.

BOD, Biochemical Oxygen Demand - Determination to measure the quantity of oxygen that is required by bacteria to oxidize, or render stable, the more easily decomposable organic substance of a waste.

BOD, 5-day Value - Amount of oxygen required over a 5-day incubation period.

BOD, 20-day Value - Amount of oxygen required over a 20-day incubation period.

Biofiltration - Process by which raw waste or sewage is passed through a bed of porous stone which is coated with active micro-organisms.

Bioxidation - Oxidation by biological action.

Catabolism - Process of changing food into tissues where heat is evolved.

COD, Chemical Oxygen Demand - Oxygen required measurement based upon the oxidation of organic materials by active oxygen liberated when potassium dichromate is brought in contact with a strong acid at high temperature.

Compost - Well decayed mixture of manure, sods, leafmold, peat or other organic matter used to fertilize land.

Composting - Act of making compost.

Culture - Cultivation of micro-organisms.

Decomposition - Breakdown of organic matter through bioxidation.

Digestion - The process by which cell fluid, protoplasm, breaks down food within the cell.

Digestor - Apparatus in which digestion takes place.

Dissimulation - Breakdown of organic matter into simple compounds and oxidized products.

Endoenzyme - Enzymes working inside the cell to break down assimulated food.

Exoenzyme - Enzymes working outside the cell to break down the waste so that it can pass through the cell membrane.

Enzyme - substances which speed up a biochemical reaction.

Fermentation - Process involving the liberation of energy and gas without the utilization of atmospheric oxygen common to anaerobic type methods.

Humus - Black organic matter formed by the decomposition of plant and animal remains.

Inocculation - voluntary infection with germs or virus of a culture medium or a living organism.

Lagooning - Process of storing wastes in shallow basins for periods ranging from one day to several months, during which time several types of purification occur.

Manure - solid and liquid waste of livestock and also well decayed organic matter.

Mesophilic - Medium temperature loving, 65° to 113°F.

Metabolism - All the chemical and physical processes of the body.

Microbiological - Process employing micro-organisms.

Micro-organism - A microscopic organism, such as a microbe.

Mutation - The first appearance of a new trait in an offspring.

Organism - The entire body of any living thing.

Phychophilic - Cold temperature loving, 68°F. or less.

Saccharification - Process of converting organic substances into sugar.

Selection - Process by which certain groups of organisms become numerous.

Superposed Decks - Decks placed one upon the other.

Thermophilic - Heat loving, 113°F. or above.

Volatile solids - That portion of organic matter which can be volatilized in a muffle furnace at 525°C.

White Water - Effluent from paper machine containing clay, starch and fibre.

XI. BIBLIOGRAPHY

(1) Adeney, W. E. and Dawson, B.B.; Sci., Pro. Roy. Dublin Soc. 18 (N.S.), 199 (1926).

(2) Beattie, J. H.; "Preparation of Potting Soil, Compost and Artificial Manure," Pamphlet from U.S. Dept. of Agri.

(3) Besselievre, E.R.; "Industrial Waste Treatment", McGraw Hill Book Company (1952)

(4) "Bioxidation of Wastes," Chem. Eng., 62, 112 (Aug. 1955).

(5) Boruff, C.S.; I. & E. Chem., 31, 1335 (1939)

(6) Bromfield, Louis, "Malabar Farm", Harper & Bro. N.Y.; 126, 174, (1948).

(7) Buswell, A.M. and Hatfield, W.D.; State Water Survey , Ill. Bull. 32 (1939).

(8) Coe, R.H.; Sewage and Ind. Wastes, 24, 731 (1952).

(9) "Custom Culture," I & E Chem., 44, 13A (Dec. 1952).

(10) DeBeeze, G. and Liebmann, A.J.; I & E Chem., 44, 882 (Oct. 1944).

(11) Dougherty, R.J.; 6th Ind. Waste Conf., Purdue Univ., Series No. 76, 479 (1951)

(12) Dunning, J.W. and Lathrop, E.C.; I & E Chem., 37, 24 (1945).

(13) Larp-Thomas,G.H.; U.S. 2,178,818 issued Nov. 7, 1939

(14) Larp-Thomas,G.H.; U.S. 2,535,627 issued Dec. 26, 1950

(15) Eckenfelder,W.W. Jr.; 7th Ind. Waste.Conf., Purdue Univ., Series No. 79, 181 (1952).

(16) Eckenfelder,W.W. Jr.; Sewage Works J., 26, 1334 (Nov, 1952).

(17) Eckenfelder,W.W.Jr.; & Grich, E.R., 10th Ind. Waste Conf., Purdue Univ., Series No. 89, 549, (1955).

(18) Eckenfelder,W.W. Jr. & Hood, J.W., 6th Ind. Waste Conf., Purdue Univ., Series No. 76, 221, (1951).

(19) Eckenfelder,W.W. Jr., & Moore, T.L.; Chem. Engr., 62, 189, (Sept. 1955).

(20) Eckenfelder,W.W. Jr., & O'Connor, D.J.; Amer. Soc. of Civil Engr., 80, Separate 411, (1954).

(21) Eweson, E.W.; U.S. 2,593,867, issued april 22, 1952.

(22) Fellenberg, Th. Von; Biochem Z., 188, 365, (1927).

(23) Gaden, E. Jr., Chem. Engr., 63, 159, (April 1956).

(24) Gainey,P.L. & Lord T.H.; "Microbiology of Water & Sewage," Pub. Burgess Publishing Co., P5-10, (1950).

(25) Gellman, I. & Heukelekian, H.; Sewage & Ind. Wastes, 23, 1267, (1951).

(26) Gilbert, F.A.; "Mineral Nutrition of Plants & Animals," Univ. of Oklahoma Press (1949).

(27) Gurnham, C.F.; "Principles of Ind. Waste Treatment," Pub. John Wiley & Sons, Inc., (1955) P. 326.

(28) Harris, E.E., Beglinger, E., Hajny, J. & Sherrard,E.C.; I & E. Chem., 37, 12, (1945).

(29) Hayek, M. & Shriner, R.L.; I. & E. Chem., 36, 1001, (1944).

(30) Helmers, E.N.,Frame, J.D., Greenberg,A.E. & Sawyer, C.N.; 6th Ind. Waste Conf., Purdue Univ.Series No.76, 375, (1951).

(31) Heukelekian,H., Ind. & Eng. Chem., 41, 1412, (July 1949)

(32) Heukelekian,H., Sewage & Ind. Wastes, 22, 87, (1950).

(33) Kominek, E.G., Chem Eng. Progress, 45, 417, (July 1949).

(34) Hood, J.W.; Sewage Works J., 20, 640, (1948).

(35) Hoover, S.R., Jasewicz, L., Pepinsky, J.B. & Porges, J.; Sewage & Ind. Wastes, 23, 167, (1951).

(36) Hoover, S.R., Jasewicz,L. & Porges, N.; 9th Ind. Waste Conf. Purdue Univ. Series No. 87, 71 (1954).

(37) Hoover, S.R. & Porges, N.; 4th Ind. Waste Conf., Purdue Univ., Series 72, 137, (1949).

(38) Hoover, S.R. & Porges N., Sewage & Ind. Wastes, 24, 306, (1952).

(39) Hoskins: Sewage Works Engr., 17, 493, (1945)

(40) Ingols, R.S. & Murray, RE.; Water & Sewage Works J., 95, 113, (1948).

(41) Jacobs, H.L.; Chem Engr., 62, 184, (April 1955).

(42) Johnson, D.W., Halvorson, H.D. & Tsuchiya, H.M.; Abstracts of 109th Meeting, Amer. Chem. Soc., Atlantic City, N. J. P. 2S, (1946).

(43) Katzen, R., Aries, R.S. & Othmer, D.F.; I. & E. Chem., 37, 442, (1945).

(44) Kaufman, S., 9th Ind. Waste Conf., Purdue Univ., Series No. 87, 107, (1954).

(45) Martin, J.P. and Waksman, S.A.; Circular 470 from N.J. Agricultural Experiment Station, October 1952.

(46) Mirkin, S.; N.Y. Daily News; P. 38, Aug. 26, 1954.

(47) Mitchell, R.D., Cassanos, J.G. & Okun, D.A.; Sewage & Ind. Wastes, 23, 1001, (1951).

(48) Moon, T.J., Mann, P.B. & Otto, J.H.; "Modern Biology", Henry Holt Pub. Co., 480, (1951).

(49) Moore, W.A.,Kroner, R.C. & Ruchhoft, C.C.; Anal. Chem., 21, 953, (1949).

(50) Niles, Jr. C.F.; 4th Ind. Waste Conf., Purdue Univ., Series 68, 80, (1949).

(51) "Official Methods of Analysis", 7th Ed., Pub. by Assoc. of Official Agricultural Chemists (1950).

(52) Oldhue, J.Y.; 10th Ind. Waste Conf., Purdue Univ., Series No. 89, 391, (1955).

(53) Parran, T.; Ind. & Eng. Chem., 39, 560, (1947).

(54) Pearson, E.A., Feuerstein, D.F. & Onodera, B.; 10th Ind. Waste Conf., Series No. 89, 334, (1955).

(55) Pepinsky, J.B., Porges, N. & Hoover, S.R.; 6th Ind. Waste Conf., Purdue Univ., Ser. No. 76, 367, (1951).

(56) Pitman, F.; Food Engr., 25, 67, (Nov. 1953).

(57) Plow, R.H., Saeman, J.F., Turner, H.D. & Sherrard, E.C., 37, 36, (1945), I. & E. Chem.

(58) Porges, N., Jasewicz, L. & Hoover, S.R.; 10th Ind. Waste Conf., Purdue Univ., Ser. No. 89, 135, (1955).

(59) Prescott, S.C. & Dunn, C.G., "Industrial Microbiology", McGraw Hill Book Co., 11, (1949).

(60) Ibid P. 245.

(61) Ibid P. 60.

(62) Rhame, G.A., Water & Sewage Work J. 94, 192, (1947).

(63) Roberts, H.V. and Sanderson, W.W.; Sewage Ind. Wastes, 25, 793, (1953).

(64) Rohlick, G.A.; Sewage Works J. 16, 540, (1944).

(65) Rudolphs, W.; Sewage & Ind. Wastes, 22, 1157, (1950).

(66) Rudolphs, W. & Amberg, H.R.; Sewage & Ind. Wastes, 25, 191, (1953).

(67) Rupp, V.R.; 6th Ind. Waste Conf., Purdue Univ., Ser. 76, 363, (1951).

(68) Russell, G.D. & Russel, G.S.; 9th Ind. Waste Conf., Purdue Univ. Ser. 87, 201, (1954).

(69) Sanborn, N.H.; Canning Trade J., 74, No. 33, 17, (1952).

(70) Sanborn, N.H.; Sewage & Ind. Wastes, 25, 1034, (1953).

(71) Sawyer, C.N.; Sewage Works J., 12, 244, (1940).

(72) Sawyer, C.N., Moore,M.C. and Weinberger, L.W., "Nitrate Nitrogen Determination", Unpub., Mass. Inst. of Tech. (1948).

(73) Schoene, L., Fulmer, E. I. & Underfofla, L.A.; 32, 544, (1940). I. & E. Chem.

(74) "Standard Methods for the Examination of Water & Sewage", 9th Ed., Amer. Pub. Health Assoc. N.Y.C., 122, (1946).

(75) "Standard Methods for the Examination of Water & Sewage", (1950) Amer. Pub. Health Assoc. N.Y.C.

(76) Stevens, W.B.; Sewage & Ind. Wastes, 24, 904, (1952).

(77) Taylor, W.D, Voegtlin, E.W., Schlosser, C.E.; U.S. 2,611,975 Issued Sept. 30, 1952.

(78) Tech. & Com. Development Reports, I. &.E. Chem., 46, Jan. to June 1954, 13A, advertising Section.

(79) Thoman, J.R., Suppl. Pub. Health Repts. No. 213, U.S. Public Health Service (1950)

(80) Ukers, W.H.; "All About Coffee", Pub. by N.Y. Tea and Coffee Trade J. Co., Chap. 15, 23 7 24, (1935).

(81) Unger, E.D., Ch. E. Thesis, Case School of Applied Science, (May 1941).

(82) U.S. 2,086,717; Austrian 150,279; German 662,720.

(83) U.S. Public Health Service, "Inventory of Water & Sewage Facilities in the United States", Cincinnati (1948).

(84) Vilbrandt, F.C., Mather, C.G. & Dicks, R.S., 32, 169, (1940). I. & E. CHEM.

(85) Wiley, J.S.; 10th Ind. Waste Conf., Purdue Univ., series No. 89, 306, (1955).

(86) Yushok, W. & Bear, F.E., Bulletin 707 from N.J. Agri. Exper. Station, (October 1948).