FRUIT FLAVORS

Apparatus and Procedure for Separation and Estimation of Volatile Components

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A vacuum distillation apparatus for the separation of volatile flavor compounds from fruit products has been developed which may be used as a flavor stripper or a concentrator for heat-sensitive substances and can be operated under a closed system at temperatures of 25° to 30° C. By use of this apparatus essentially all the volatiles from fruit samples may be removed without alteration due to heat damage such as occurs during steam distillation at atmospheric pressure. The procedure adopted gives fairly reproducible results on a variety of fruits.

CHANGES IN THE VOLATILE FLAVOR FRACTION of certain frozen fruits were investigated during the course of work on flavor stability. Most existing methods for the determination of volatile compounds in food products make use of steam distillation at atmospheric pressure. Although this procedure is satisfactory for many purposes, there is always the danger of altering certain heat-sensitive compounds by employing relatively high distilling temperatures. The authors were interested in obtaining the naturally occurring volatiles with as little alteration in composition as possible.

Lang *et al.* have described a method for assessing the quality of fish by the chemical evaluation of the odorous or volatile compounds derived from the product (*4*). Farber presented data which demonstrated the applicability of this procedure to other food products (*2*). The method involves aeration of an aqueous sample with a measured volume of clean air, which is subsequently passed through two flasks of alkaline permanganate solution. The volatiles in the sample are measured by the amount of permanganate reduced. While this procedure eliminates the objection of operating at high temperatures, only a fraction of the total volatiles from fruit samples is removed.

In view of the limitations of the aeration technique, it was decided to design a low-temperature vacuum steam-distillation unit which would give reproducible results and a good recovery of the volatile flavors from fruits or fruit products. Although parts of this unit are similar to those of the vacuum flash evaporator described by Dimick and Makower (*1*), certain features were added which make the unit more versatile and adaptable to the separation of volatiles from small samples.

Apparatus

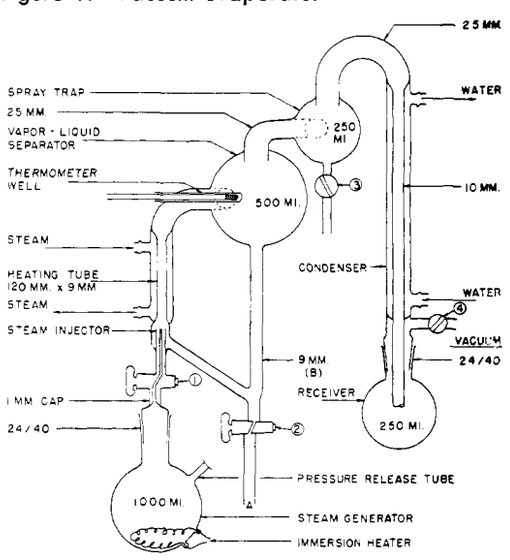
The all-glass apparatus shown in Figure 1 consists of a steam generator, steam-injection jet, steam-jacketed heating tube, cyclone vapor-liquid separator, spray trap, straight-tube condenser, and receiver. With the exception of the steam generator and receiver, the apparatus is preferably built as a single unit. The steam generator is heated with a Nichrome immersion heater, which is controlled by a variable transformer. Steam is admitted to the system through a 1-mm. injector, which aids in the recirculation of the material to be stripped. Tube *B*, which connects the vapor-liquid separator and the steam-jacketed heating section, provides a path for continuous recirculation of the sample, which is introduced at inlet *A*. The vapor tubes enter the vapor-liquid separator and the spray trap tangentially; this type of construction reduces the possibility of the samples frothing over into the distillate.

Vacuum is applied to the system by means of a mechanical pump through an inlet which is built into the condenser between the lower end of the water jacket and ground-glass joint. During the distillation a three-way stopcock, No. 4, isolates the system from the pump and thus prevents loss of volatiles to the vacuum source. In order to minimize air leaks into the system, all stopcocks are oblique bore.

Procedure

Because the evaporator is constructed essentially in one piece to minimize the possibility of leaks, only the steam generator and receiver require connection prior to operation. With stopcocks 1 and 4 open and 2 and 3 closed, the entire apparatus is evacuated to a pressure of approximately 5 mm. of mercury by means of a mechanical pump. The steam generator is quickly brought to a boil by regulation of current in the immersion heater with a variable transformer. Sufficient water is introduced at *A* to fill the tube below stopcock 2. The vacuum source is then closed by means of stopcock 4 and the sample is slowly introduced from a beaker or other suitable container through stopcock 2. Distilled water is used to flush the remaining sample, between inlet tube and stopcock 2 into the evaporator. Care should be taken to prevent air entrainment during the introduction and flushing of the sample into the system, as this would cause an increase in pressure within the closed system and hence increase the distilling temperature. Heat to the steam generator is regulated so that the distilling temperature is maintained between 25° and 30° C. If there are no air leaks in the ground-glass joints or stopcocks,

Figure 1. Vacuum evaporator



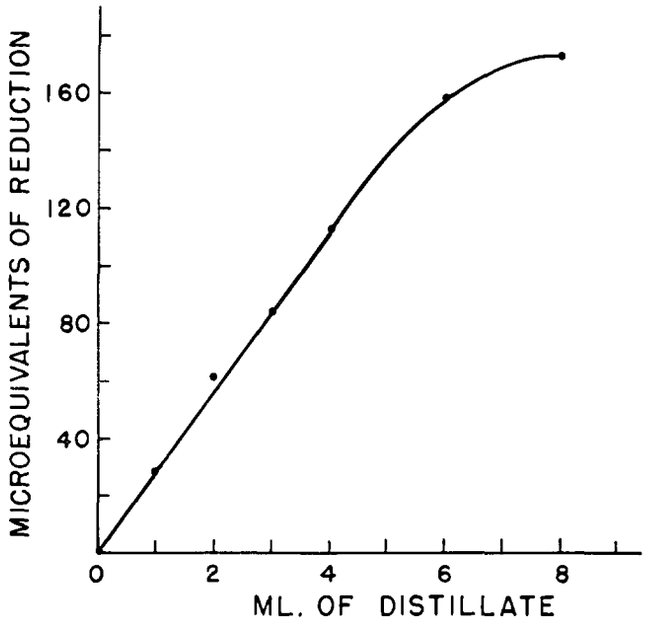


Figure 2. Relation of permanganate reduction to amount of strawberry volatiles

distillation under these conditions can be continued until any desired volume of distillate is collected. During distillation, the receiver is immersed in an ice-water bath.

If it is desired to concentrate the sample in addition to stripping off the volatiles, stopcock 1 is closed, and the steam-jacketed heating tube is used as the sole heating source. The sample can be concentrated to a volume of 15 to 20 ml. If further stripping is desired, additional distilled water is admitted through No. 2 and the distillation is allowed to proceed as before.

After the desired amount of distillate has been collected, all heating units are turned off and stopcock 1 is closed. The system is brought to atmospheric pressure by slowly admitting air through stopcock 3. The stripped sample is removed by opening stopcock 2 and allowing the material to drain. The apparatus is cleaned by circulation of water through the system and rinsing with distilled water.

Analytical Method

The method of Friedemann and Klaas (*3*) was employed to obtain a measure of the total volatiles in the distillates collected in the receiver. This method depends on the oxidation of the volatiles by 5 ml. of 0.1*N* potassium permanganate in 1.0*N* sodium hydroxide, and the results are expressed as microequivalents of reduction. In order to determine the range and reproducibility of the method in measuring fruit volatiles, aliquots of a single distillate from Marshall strawberry puree were analyzed for total volatiles. Figure 2 shows an essentially linear relationship between the amount of distillate and microequivalents of reduction within the range of 0 to 120 microequivalents. Consequently, all subsequent measurements were made within this range.

Results

Operating at temperatures of 25° to 30° C., the evaporation rate is approximately 2 ml. of distillate per minute.

In order to determine the relationship between the amount of distillate collected and recovery of volatiles, aliquots of grape juice and cherry puree were stripped for varying times. As shown in Table I, recovery of the volatiles from fruit samples approaches a maximum value when the amount of distillate collected is approximately twice that of the sample introduced. As the apparatus was primarily designed for the estimation of volatiles in small samples, the sample size was usually limited to 20 to 30 grams. Therefore one distillation required from 20 to 30 minutes, depending on sample size.

Table I. Flavor Stripping Efficiency on Different Fruits

Fruit	Ratio of Vol. of Distillate to Wt. of Sample	Microequivalents of Reduction per Gram of Sample
Grape juice	0.9	113
	1.3	119
	3.4	124
Cherry puree	0.8	76
	1.5	80
	4.4	82

Total volatiles were determined on four replicate samples of grape juice and duplicate samples of peach, strawberry, and cherry puree. The results expressed as microequivalents of reduction per gram of sample were as follows: grape juice, 65, 65, 64, 63; peach puree, 27, 28; strawberry puree, 17, 18; cherry puree, 128, 131. These data indicate that this procedure gives fairly reproducible results on a variety of fruits. It must be emphasized, however, that in evaluating the reliability of an apparatus and procedure for volatiles in fruit samples, the duplicate determinations must be made on a uniformly homogenized sample of tissue. Otherwise, discrepancies may occur which are actually due to variation of the volatiles in the fruit and not to errors in procedure or methods.

Examples of typical values obtained from various frozen fruits (Table II) clearly show the large variations possible in fruit of the same variety. As most of these samples were obtained from commercial plants in various sections of the West Coast, the variations are probably due to differences in growing areas, cultural conditions, and processing procedures.

Table II. Typical Examples of Amounts of Volatiles Found in Commercial Samples of Frozen Fruit

Fruit and Variety	Code	Microequivalents of Reduction per Gram
Marshall strawberry	B II	11
	B I	13
	C II	15
	C I	18
	B D	20
	L	23
Hybrid strawberry	J	23
Bing cherries	I	21
	II	31
	III	37
Montmorency cherries	AL	68
	BX	104
	CM	122
	DT	134
J. H. Hale peaches	A	28
	B	53
Kirkman Gem peaches	...	48
Fay Elberta peaches	...	28
Elberta peaches	5	24
	7	40
	1	60

While the apparatus has been primarily used for the separation of volatile flavors from fruits and other food products, it is also valuable as a concentrator for heat-sensitive substances. At this laboratory, it has been used to concentrate heat-labile fruit enzymes and other biological materials.

Literature Cited

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