

FRUIT CONCENTRATES

Flavor-Fortified High-Density Frozen Citrus Concentrates

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Full-flavored, high-density frozen orange juice and grapefruit juice concentrates were prepared successfully by adding appropriate amounts of cold-pressed peel oil to juice concentrates of the desired level of concentration. Packs of different concentrations from fourfold to sevenfold were prepared and their storage behavior was studied at -18°C . (0°F .), -7°C . (20°F .), and 5°C . (40°F .). Flavor and cloud stability at -7°C . (20°F .) and 5°C . (40°F .) increased with increasing concentration of the juice above fourfold. The degree of cloud stability obtained at the sixfold level of concentration was equal to that obtained by stabilization of fourfold concentrates by partial heat treatment. In addition to savings in refrigeration costs from reduced bulk and lower freezing point, large size containers of sixfold concentrates were more easily reconstituted than such packs of the standard fourfold product, because they were more fluid at the usual frozen storage temperatures.

FULL-FLAVORED HIGH-DENSITY FROZEN CITRUS CONCENTRATES can be prepared by fortification with peel oil (8). Recent developments in the field of low temperature evaporation have made possible the production of higher density products by the cutback process (6) used commercially for producing frozen citrus concentrates. In view of the work of Cotton *et al.* (2) suggesting that juice cloud stability in orange concentrates tended to increase with increasing concentration above 42° Brix and the work of Rouse (70) indicating that the pectase activity of orange concentrate decreased above 42° Brix, it appeared that high-density frozen citrus concentrates would possess definite advantages in storage stability over 42° Brix (fourfold) products.

This paper reports studies on the preparation and comparative storage stabilities of four-, five-, six-, and sevenfold orange and grapefruit concentrates prepared by peel oil fortification.

Experimental Work

Juice packs of different concentrations from fourfold to sevenfold, flavor fortified with equivalent amounts of peel oil (on a reconstituted basis), were prepared. In most cases commercial sixfold concentrate served as the raw material, dilutions being made with distilled water or further concentration being obtained in a pilot plant low-temperature falling-film evaporator.

Samples Prepared

Twenty-six packs of California Valencia orange juice concentrate without added sugar were prepared from commercial sixfold concentrate obtained from California plants during the 1951 and 1952 seasons. These included 12 packs of fourfold concentrate prepared by diluting the commercial sixfold product with distilled water and adding peel oil, one pack of fivefold concentrate prepared similarly, and 11 packs of sixfold concentrate prepared by simply adding appropriate amounts of peel oil to the sixfold commercial concentrate. Two packs of sevenfold concentrate with added peel oil were prepared by concentrating the commercial sixfold product to sevenfold at 16°C . (60°F .).

Three packs of Valencia orange juice concentrate were prepared from fresh fruit obtained during the middle of the 1951 season. This fruit was juiced manually, screened through a 30-mesh sieve, and evaporated in a pilot plant low-temperature evaporator. One batch of juice was evaporated to fourfold concentration and fortified with peel oil and two batches of juice were evaporated to sixfold and fortified with peel oil.

Two packs of Marsh Seedless grapefruit juice concentrate without added sugar were prepared from a commercial fourfold product obtained from a California plant during the 1952 season. The commercial fourfold product was concentrated to sixfold in a pilot plant low-temperature evaporator. One pack of

peel oil-fortified sixfold concentrate was prepared from part of the lot; the remainder was diluted back to fourfold concentration with distilled water and an appropriate amount of peel oil was added. Juice from which the original concentrate was produced had been heat-treated for 1 second at about 66°C . (150°F .).

Peel oil was added by mixing with a portion of the batch of concentrate being formulated, blending the mixture with the rest of the batch, and filling into cans. Peel oil does not mix well with single-strength juice but blends easily enough with small batches of concentrated juice, so that the mixture does not have to be stirred while cans are being filled (Table I).

Each pack of product consisted of 24 to 48 6-ounce cans. These packs were stored at -18°C . (0°F .), -7°C . (20°F .), and 5°C . (40°F .) and analyzed periodically for cloud loss and rate of flavor change. The products were prepared for analysis by reconstituting them to juices of 12° Brix by adding water; the resulting juices averaged ap-

Table I. Distribution of Added Oil in Orange Concentrate

No. of Cans Filled Consecutively	Recoverable Oil	
	Series A (unstirred)	Series B (stirred)
1 and 2	0.012	0.012
9 and 11	0.011	0.012
19 and 20	0.013	0.013

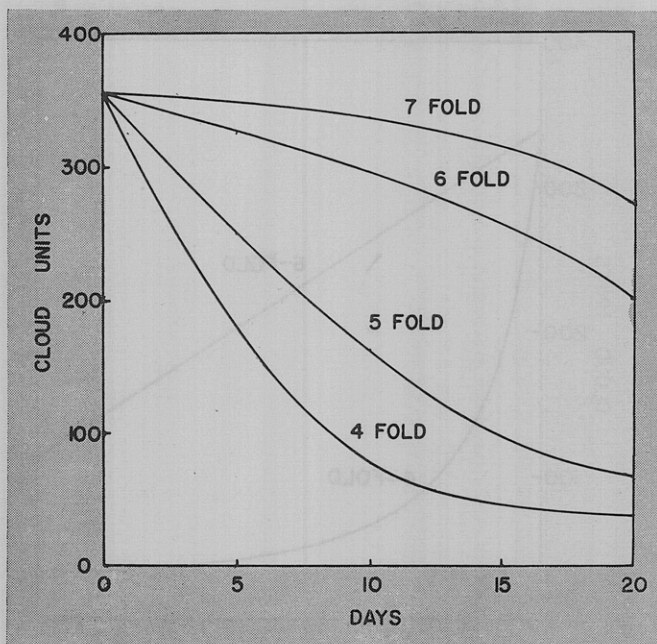


Figure 1. Cloud stability of orange juice concentrate at 20° F.

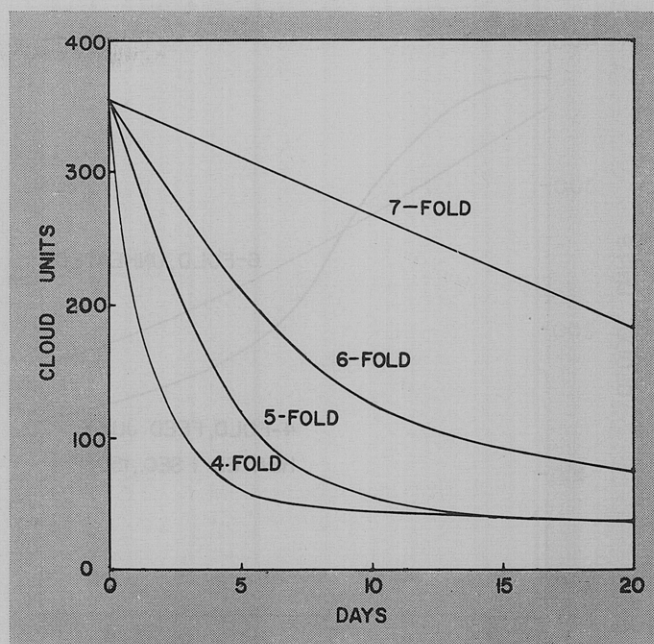


Figure 2. Cloud stability of orange juice concentrate at 40° F.

proximately 6% pulp. The pH range of the reconstituted orange juices was 3.5 to 3.6 and the total acidity was 1.10 to 1.19% (gram/gram). The pH of the reconstituted grapefruit juice was 3.6.

Juice cloud stability was determined by a method similar to Loeffler's (5). The juice was centrifuged for 15 minutes at 2000 r.p.m. and the transmittance of the supernatant measured in a Klett colorimeter using a No. 66 red filter. The light transmittance value as measured was termed the cloud unit.

Flavor Determination

At the beginning of the storage tests, taste tests were run on the packs of juice of varying concentration to make sure that the preparation of such products alone did not cause any flavor changes in the original juice. The rate of flavor change for each product was determined by comparing the stored product periodically with some of the product kept at -18° C. (0° F.).

Flavor changes were measured by the triangular taste-testing method for determining flavor differences (1, 4, 7). The taste panel developed for these studies consisted of 10 to 12 people whose performance on a series of samples of selected difficulty was 55% correct choices or better. Using 20 judgments per test (10 people duplicating their test at each sitting), the storage was arbitrarily terminated when a significant difference in flavor could be found and duplicated. The statistical level of significance which was considered to demonstrate a difference was 5 to 1%, or 11 to 13 correct judgments out of a total of 20 (5% = 19 to 1 odds and 1% = 99 to 1 odds that the results were not due to chance).

Results and Discussion

Cloud Stability

The most striking difference between the fourfold products and those of higher concentration was in cloud stability during storage. Figures 1 and 2 show the typical effect of concentration on cloud stability in a series of orange concentrates from the same batch of juice over the range of fourfold to sevenfold. The only differences in behavior between series of juice concentrate packs were in absolute cloud values and absolute rates of cloud change. The superior cloud

stability of higher-density products was clearly evident in all cases.

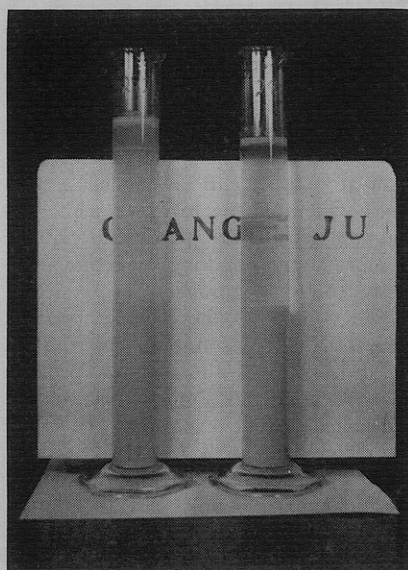
Figure 3 offers a photographic comparison of the difference in cloud stability of a fourfold product after storage for 20 days at -7° C. (20° F.) with a sixfold product after storage for 41 days at the same temperature. The graduated cylinders contain samples of the reconstituted juices allowed to stand 4 hours at room temperature before photographing. In general, cloud loss was considered objectionable when the cloud values approached 100. Objectionable change in cloud usually preceded detectable flavor changes.

A contemporary industrial practice for increasing the cloud stability of fourfold citrus concentrates is to heat the juice at some stage during its concentration. Some plants heat the evaporator feed juice and some the concentrated juice at one of the stages in the multiple-stage evaporator during the concentration step. This heat treatment inactivates some of the pectic enzymes in the juice and increases the cloud stability of the resulting product. Figure 4 compares the cloud stability of a commercial fourfold orange concentrate, prepared by heating the evaporator feed juice to about 66° C. (150° F.) for 1 second, with an experimental unheated sixfold product prepared in the pilot plant. It appears from Figure 4 that packing concentrate as a sixfold product achieved about the same cloud stabilization as was obtained by heating the feed juice of the fourfold product under the conditions cited.

As long as appreciable amounts of the enzymes causing cloud loss in orange and grapefruit juice remain active after

Figure 3. Cloud stability of orange juice concentrate at 20° F.

Left. Sixfold product stored 41 days at 20° F. (125 cloud units)
 Right. Fourfold product stored 20 days at 20° F. (43 cloud units)



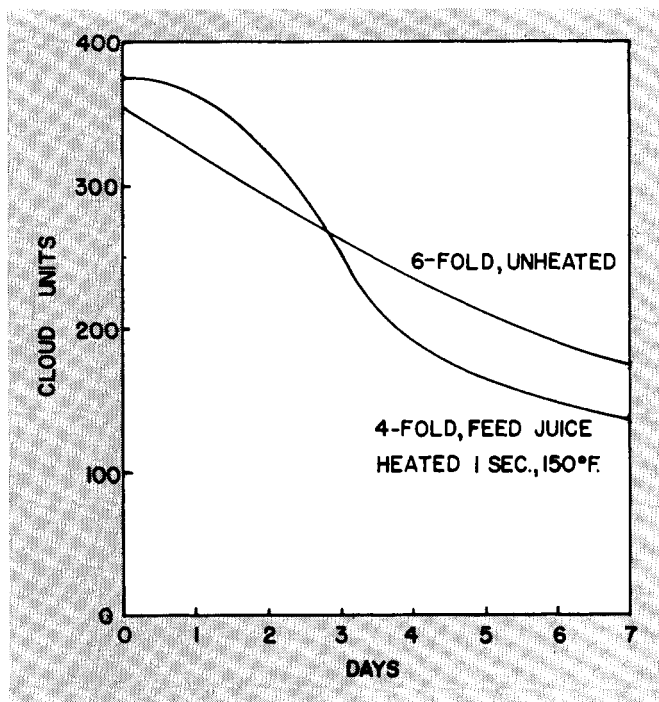


Figure 4. Cloud stability of fourfold heated and sixfold unheated orange juice concentrate at 40° F.

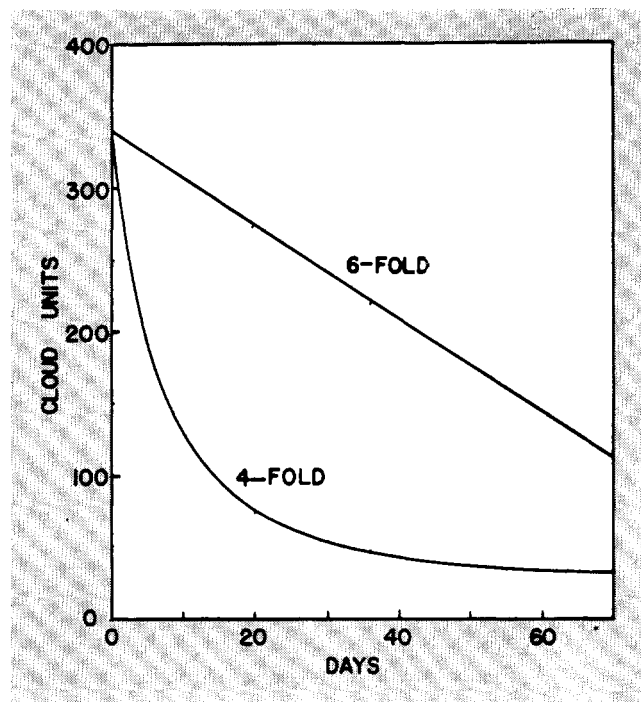


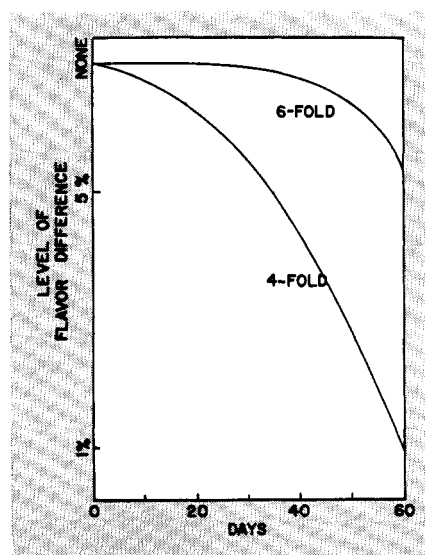
Figure 5. Cloud stability of heat-treated grapefruit juice concentrate at 40° F.

heating, the cloud stability of such heated products could be further improved by packing the juice as a sixfold concentrate rather than as the fourfold product (Figure 5).

Flavor Stability An additional benefit typical of these studies was that flavor stability tended to increase with juice concentration under these conditions. As can be seen in Figure 6, this conclusion was based upon measuring the rates at which flavor dif-

Figure 6. Rate of flavor change in orange concentrate stored at 20° F.

Numbers in ordinate represent statistical significance of flavor differences observed between control sample and stored sample



ferences in the product approached a given level of statistical significance. This method of determining product storage stability was a more objective one than trying to determine the time necessary for a product to become "unacceptable." Because members of the taste panel used for this study were a selected group trained to detect differences in citrus juices, the results presented show a picture of flavor difference perception which would be considered better than average.

No difference in flavor stability was established in the grapefruit juice concentrate series.

In addition to improved cloud and flavor stability, the sixfold products have certain other desirable physical properties.

They do not become so solid on cooling to -18° C. (0° F.) as the standard fourfold product, a feature of interest to those handling institutional-size containers of concentrate. For example, in gallon-size containers, fourfold concentrate at -18° C. (0° F.) forms a solid block which is slow to thaw, while sixfold concentrates are plastic and capable of more rapid handling on removal directly from cold storage.

The volume of the fourfold product increases about 5% on cooling to -18° C. (0° F.), whereas the sixfold product shows no appreciable volume change (3). This means that cans may be more completely filled with the sixfold product, since less allowance need be made for product expansion on cooling to -18° C.

The sixfold concentrates begin to freeze at approximately -15° C. (5° F.)

and contain about 2.5% ice at -18° C. (0° F.) while the fourfold ones begin to freeze at approximately -8° C. (17.6° F.) and contain 32% ice at -18° C. (0° F.) (9). Because of this difference in freezing point and the obvious difference in bulk between the sixfold and fourfold products, there should be savings in refrigeration costs.

Literature Cited

- (1) Byer, A. J., and Abrams, Dorothy, *Food Technol.*, **7**, 185-7 (1953).
- (2) Cotton, R. H., Roy, W. R., Brokow, C. H., McDuff, O. R., and Schroeder, A. L., *Proc. Florida State Hort. Soc.*, **60**, 39 (1947).
- (3) Joslyn, M. A., and Marsh, G. L., College of Agr., Univ. California, Berkeley, *Bull.* **551**, 16 (May 1933).
- (4) Kramer, Amihud, *Food Eng.*, **24**, 100-8 (December, 1952).
- (5) Loeffler, H. J., *Ind. Eng. Chem.*, **33**, 1308 (1941).
- (6) MacDowell, L. G., Moore, E. I., and Atkinson, C. G., U. S. Patent 2,453,109 (1948).
- (7) Peryam, D. R., and Swartz, V. W., *Food Technol.*, **4**, 390 (1950).
- (8) Rice, R. G., Keller, G. J., and Beavens, E. A., *Ibid.*, **6**, 35 (1952).
- (9) Riedel, L., *Kältetechnik*, **2**, No. 8, 201 (1950).
- (10) Rouse, A. H., *Citrus Ind.*, **31**, 7 (1950).

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