Odor in Refined Sugar

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One of the most precise methods for quantitative evaluation of odor is the threshold procedure. The threshold number represents the extent to which an odor-bearing sample must be diluted with odor-free water in order to make its odor barely perceptible. The threshold-odor test used for many years in the water treatment field was modified by the Suchar Sales Corporation for sugar work and published in *Sugar Journal,* November 1961. In applying this procedure to the analysis of refined beet sugars, it soon became evident that the method lacked sufficient sensitivity for our purpose. A modification of this Suchar method, however, was developed which shows considerable promise. For the past operating season this method has been used to determine threshold odor numbers on daily composite sugar samples from the six Utah-Idaho Sugar Company factories.

Procedure

The test with our modifications follows:

Reagents and Equipment

Odor-free (carbon treated) water.
C.P. citric acid, 10% solution in odor-free water.
Six 500 ml glass-stoppered Erlenmeyer flasks.
Water bath controlled at 60° C.

Determination

Dissolve 200 gm of the sugar to be examined in 200 ml of odor-free water at room temperature. This provides the test solution (Solution A) which will be used for further dilution. Table 1 shows the aliquots of this odor-bearing Solution A to be added to freshly rinsed flasks for the determination of the odor range.

Table 1.—Milliliters of solution A to be used in first dilution to determine range.

Flask No.	Milliliters
1	200
2	50
3	12
4	2.8

Dilute each sample to 200 ml with odor-free water and add 5 ml of 10% citric acid solution. Also add 200 ml of odor-

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free water plus 5 ml of 10% citric acid solution to another flask as a reference. Heat all flasks to 60° C in a hot-water bath.

Shake the flask containing the reference solution, remove the stopper, and sniff the vapors. Replace the stopper. Do the same with the flask containing the 200 ml of sample under test and observe whether by comparison it contains an odor differing from the slight characteristic odor of the citric acid. Repeat this procedure with the flask containing the next lower concentration, always sniffing the reference solution first. Record which flasks present differences and which do not. Based on the results of the preliminary test prepare one of the following sets of dilutions:

Range 1: Difference with 200 ml but not with 50 ml
Range 2: Difference with 50 ml but not with 12 ml
Range 3: Difference with 12 ml but not with 2.8 ml
Range 4: Difference with 2.8 ml.

Table 2 shows the dilutions of Solution A to be used with each of these four ranges.

Table 2.—Milliliters of solution A to be used for determination of threshold odor number.

Range number	Flask number	Milliliters	Range number	Flask number	Milliliters
1	1	200	2	1	50
	2	140		2	35
	3	100		3	25
	4	70		4	17
	5	50		5	12
3	1	12	4	1	2.8
	2	8.3		2	2.0
	3	5.7		3	1.4
	4	4.0		4	1.0
	5	2.8			

If necessary, make up another Solution A and proceed as described previously diluting each to 200 ml, adding citric acid solution, and heating to 60° C. Arrange the flasks so that their identity is unknown, and compare each with the reference solution. Place the flasks in which odor difference was observed in one group and those without odor difference in another. The dilution containing the smallest volume of sample which gives a positive test determines the threshold odor. By reference to Table 3 the corresponding threshold number is determined.

Table 3.—Dilution conversion table.

Milliliters of solution A	Threshold number	Milliliters of solution A	Threshold number
200	1	12	17
140	1.4	8.3	24
100	2	5.7	35
70	3	4.0	50
50	4	2.8	70
35	6	2.0	100
25	8	1.4	140
17	12	1.0	200

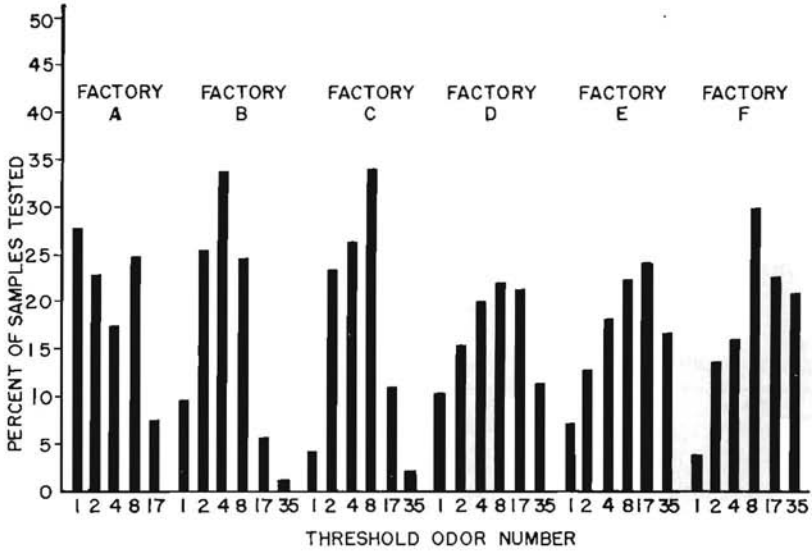

Figure 1.—Results of testing approximately 100 days of production at six factories during 1963.

To obtain consistent results in odor measurements, it is necessary that the analyst always observe a technique somewhat as follows:

1. Some practice is necessary to develop consistent threshold sensitivity. This consistency can be readily developed in most individuals. An acute sense of smell is not essential.

2. A reliable and adequate supply of odor-free (activated carbon treated) water is required.

3. All glassware must be clean and free from odor. It is necessary to rinse all glassware several times with odor-free water prior to each test and between dilutions.

4. Tests should be run in a room as free from foreign odors as possible.

5. Each dilution should be compared with the reference solution to check judgment and minimize odor memory.

6. Care should be taken never to declare a dilution as positive in odor unless there is sufficient odor present to justify this declaration.

The results of testing approximately 100 days of production of sugar at each of our six operating factories during 1963 are shown in Figure 1.

To simplify presentation, the results have been grouped so that the results for threshold number 1 include values of 0 and 1, while those for threshold number 2 include values greater than 1 but not greater than 2, etc. It is quite apparent that there is a characteristic pattern for each factory, though the differences are more obvious when presented as in Figure 2. Figure 2 shows for each factory the percentage of sugar samples tested which would pass a theoretical threshold odor number specification.

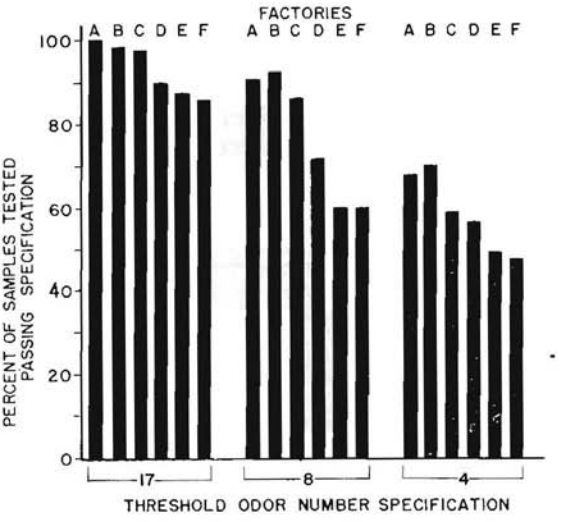

Figure 2.—Percentage of sugar samples tested which would pass a theoretical threshold odor number.

At this date we have found only one positive correlation between odor and operating conditions at the six factories. In every case, as the campaign progressed and as storage periods increased, under the same average operating conditions, the odor in sugar increased in direct proportion. Figure 3 illustrates this and while

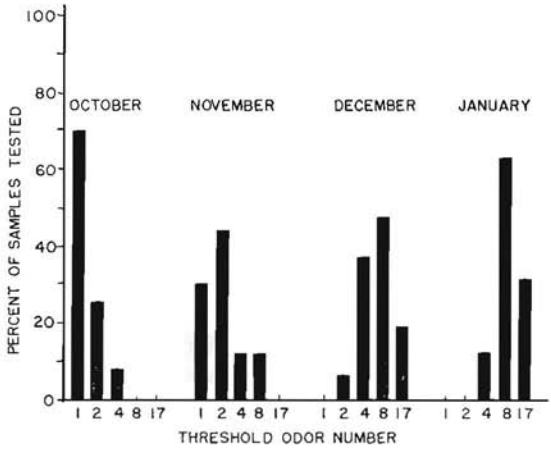

Figure 3.—One factory results for correlation between odor and operating conditions.

the data shown are taken from results of only one factory, the other five factories show similar results.

If there are other correlations between odor and operating conditions or between odor and other quality factors normally associated with refined sugar, we believe it will be necessary to obtain and analyze more data than were available at the time of this writing. There is some evidence that there are others and we hope to continue this work in an effort to demonstrate other correlations.