

INSTRUMENTATION IN THE FOOD AND BEVERAGE INDUSTRY VOLUME 1

**VOLUME 1
1972**

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**VOLUME 1
1972**



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Instrument Society of America
400 Stanwix Street
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Library of Congress Catalog Card No. 72-90018
ISBN 87664-194-X

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400 Stanwix Street
Pittsburgh, Pennsylvania 15222

FOREWORD

The food processing industry is today faced with its greatest challenge: how to improve quality while maintaining a stable price level. Those of us knowledgeable in the field of instrumentation and process control have seen tremendous benefits accrue to other process industries from the proper use and application of even the most fundamental control systems. In an effort to spread this knowledge and its resultant benefits, the Food Industry Division of the Instrument Society of America programmed an instrumentation symposium in conjunction with the Montreal Chapter of ISA. Without question, the papers presented were excellent. The message they bring is quite clear: great strides are being made in the food industry to insure better products for all consumers, but there is still a long way to go. Even though there are ultra-sophisticated computer-controlled processes in operation in the food industry, the majority of food and beverage plants are still operated manually with only a minimum of instrumentation. The profit squeeze, coupled with more stringent government regulations, will cause more and more companies to close their doors through failure, or lose their identity through merger unless they make some of the investments in equipment and manpower described in these proceedings. Consumerism, too, demands consistent quality and product purity, attainable only through automatic control systems. We hope that an exchange of ideas such as this symposium will protect the consumer, promote growth, prevent failure and insure a healthy industry.

Thanks must be extended to many people for the success of the First Annual Food and Beverage Instrumentation Symposium. The Montreal chapter of the Instrument Society of America spent many long hours planning and handling details. Session Developers Lou Gilde (Campbell Soup Co.), Terry Diebold (General Mills, Inc.) and Oscar Soroko (Fischer & Porter Co.) worked diligently at getting speakers lined up and running their sessions. But above all, Ed Nobrega (Foxboro Co.), Program Chairman of the Symposium, deserves a large share of the credit for the quality of the program. These Proceedings are, to a large measure, the result of his efforts. Our thanks to Ed for an excellent job.

A. C. Papaloannou
Director, Food Industry Div.
Instrument Society of America

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GAS CHROMATOGRAPHIC SEPARATION OF LACTOSE
AND SUCROSE AS THE TRIMETHYLSILYL DERIVATIVES

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ABSTRACT

Gas Chromatography of the trimethylsilyl (TMS) ethers of sugars is an important analytical tool in quality control laboratories of food companies. Even in TMS derivatives, the relatively non-polar high-temperature stationary phases such as SE-30 and SE-52 fail to yield satisfactory separation of α -lactose and sucrose. By the method described in this paper, the sample is dissolved in pyridine and treated with hexamethyldisilazane and trimethylchlorosilane at room temperature to convert all OH groups to TMS ethers. The derivatized sample is injected at a port temperature of 270°C. for GLC analysis into a column consisting of a section of 1.5% XE -60 on 100/120 mesh Chromosorb G followed by a section of 3% SE-52 on 70/80 mesh Anakrom ABS. The column is held at 175°C. until all monosaccharides are eluted, then the temperature is elevated 10°C./minute until 210°C. is reached where the temperature is held for the remainder of the run. Sorbitol is used as an internal standard. Excellent separation and quantitation of α -lactose and sucrose is achieved in food products such as ice cream, cake mixes, and biscuit mixes.

INTRODUCTION

The growing awareness of the differences in nutritional effects of various sugars has created an increasing need for information on the amounts of these sugars in foods. Most carbohydrate data are obtained by difference after analysis for fat, ash, protein, and moisture. Methodology problems have limited the data on individual sugars. Most published methods for specific sugars are chromatographic, including column, paper, thin-layer, and ion-exchange chromatography. All of these methods require colorimetric estimations on separated fractions and are often excessively time-consuming, insensitive, or non-specific. More recently, gas-liquid chromatography (GLC) has been adapted to the separation of individual sugars. Since McInnes and co-workers (1) discovered that methyl ethers of the methyl

glycosides of heat-labile monosaccharides were sufficiently stable and volatile to be analyzed by gas-liquid chromatography, many modifications and improvements in technique have been published. Columns containing low concentrations of liquid phases (SE-30 or QF-1) were used (2) to extend the useful range for carbohydrates to the separation of two disaccharides as octaacetates. In a similar way, Bishop (3) separated several fully methylated disaccharides.

After a rapid quantitative method for the preparation of O-trimethylsilyl derivatives was worked out by Makita and Wells (4), Sweeley *et al* (5) described a simple and rapid method for preparing trimethylsilyl (TMS) derivatives of sugars. Rickey and co-workers (6) indicated that variability resulted during quantitative determination of carbohydrates due to poor solubility in pyridine and that multiple peaks resulted from formation of anomers. In some cases, the latter factor has led to undue complexity of chromatograms from overlapping peaks. To prevent the problem of anomerization, Savardekar *et al* (7) converted sugars to their alditols and chromatographed the sugars as acetates. Tautomerization was minimized by Mason and Slever (8) when they converted sugars to oximes and then chromatographed TMS derivatives. Other workers have reported a number of GLC methods for sugar analysis in various types of foods and beverages (9-18). It was the feeling in our research laboratories that even these more refined methods for GLC analysis of sugars were too time-consuming and complex for convenient routine analyses in quality control in the food and related industries. The need for rapid and accurate lactose determination in food products has been a reality for several years. The relatively non-polar high-temperature stationary phases such as SE-30 and SE-52 fail to give a good separation of α -lactose and sucrose without involved procedures. Sweeley *et al* (5) reported retention times (relative to glucose) of 10.4 for sucrose and 10.5 for lactose on a 6 foot x $\frac{1}{8}$ inch O.D. column packed with 3% SE-52. This is not a

large enough difference to give complete resolution of these compounds within a reasonable analysis time. In our hands, α -lactose and sucrose eluted as one symmetrical peak when using an SE-52 column. The more polar polyester phases such as DEGS or EGS are generally not stable at temperatures required to elute disaccharides within a reasonable length of time. Recently, support-coated open tubular columns have been reported to separate sucrose and lactose (19) effectively; however, these columns are difficult to prepare and expensive to purchase.

With a need for quick and accurate measurement of milk ingredients as a process control measure in monitoring the addition to a variety of food products, we developed a quick and direct approach to the formation of TMS derivatives of sugars and subsequent GLC analysis. The approach described in this paper is being routinely applied to the quantitative determination of the major mono- and di-saccharides occurring in a wide variety of food preparations. These sugars include α -lactose and sucrose.

EXPERIMENTAL

In contrast to the time-consuming derivatization methods described in most of the previous literature, TMS derivatives of most of the samples that we encounter in food products can be quantitatively prepared simply by adding pyridine, hexamethyldisilazane (HMDS), and trimethylchlorosilane (TMCS) to the "raw" sample. The trimethylsilyl derivatives are formed within 15 minutes after dissolution of the sugars. An ultrasonic bath is convenient to use for rapid dissolution of sample. Our work indicates quantitative formation of TMS ethers easily within one-half hour of reagent addition if the sample vial is immersed in an ultrasonic bath for 1-2 minutes after reagents are added. Gas chromatography of the derivatives takes 50-60 minutes.

Specifically, we prepared TMS derivatives by a modification of the method of Sweeley and co-workers (5). An aliquot of product sample containing 10-20 mg of sugars and not more than 25 mg of water, plus 5-7 mg of sorbitol as internal standard, was weighed into a small vial with a teflon-lined screw cap. One ml of pyridine, 0.6 ml of HMDS, and 0.3 ml of TMCS were added in that order, and the sample was subjected to 1-2 minutes of ultrasonic treatment. After one-half hour, a 0.5-1.5 μ l aliquot of the supernate was injected for GLC analysis.

The key to getting good separation of α -lactose and sucrose in our work is the fact that we used a double-section column. The first section was a 20 foot x 1/8 inch O.D. aluminum tube packed with 1.5% XE-60 on 100/200 mesh Chromosorb G (acid washed, DMCS treated). The second section was a 7 foot x 1/8 inch O.D. aluminum tube packed with 3% SE-52 on 70/80 mesh Anakrom ABS. The two column sections

were connected with a Swagelok union. The GLC instrument used was a Research Specialties Model 1670 equipped with a dual hydrogen flame detector. The injection port temperature was 270°C. and the detector temperature was 255°C. The helium carrier gas flow rate was 20 ml/minute. Column temperature was held at 175°C. until all of the monosaccharides were eluted (16 minutes), and then programmed at 10°C./minute to 210°C. and held until the end of the run.

Sorbitol was chosen as an internal standard because it is absent from nearly all samples analyzed in our laboratory. Sorbitol does not separate well from α -glucose on XE-60 alone, but good separation is attained by using the XE-60 and SE-52 combination.

RESULTS

Figure 1 illustrates the separation of some of the common sugars found in commercial food preparations. The excellent resolution may be noted, particularly that of sucrose and α -lactose. In our experience with food products, the anomeric forms of sugars have not led to confusing overlap of peaks. We consider the elucidation of the anomeric forms of sugars in foods to be helpful information.

The addition of a large excess of HMDS and TMCS permits the direct analysis of samples containing fairly large amounts of water (15). Figure 2 is a chromatogram obtained from the direct trimethylsilylation of a 40 mg sample of ice cream, which contains about 25 mg of water. No spurious peaks due to incomplete derivatization are present. The sucrose and α -lactose are well separated, which is essential for a complete analysis of the sugars in this sample.

Table 1 shows the total sugar content of the ice cream sample as determined by GLC compared to a normal ferricyanide analysis. The data are in good agreement. The time required for analysis is approximately equal for both methods; however, the GLC approach reveals individual sugars.

Total sugars in a cake mix sample were determined by the GLC and ferricyanide methods with 41.2% and 41.3% resulting, respectively.

Since the content of lactose in milk is relatively constant, the quantitation of lactose by GLC allows the measurement of milk solids in products. GLC analysis of a sample of buttermilk powder revealed 51.6% lactose compared to a literature value of 48% (20). Table 2 reveals the percentage buttermilk solids in biscuit mix as calculated from GLC lactose values. The data indicate good agreement between milk solids calculated from lactose measurement and the known levels of addition. The GLC approach offers a quick and relatively accurate means of monitoring milk solids content

by quality control personnel as well as a good way to quickly analyze competitor products.

The sugars commonly encountered in our food products are fructose, glucose, sucrose, lactose, and maltose. The column described in this paper gives a good separation of all of them. In some instances, we obtain three anomeric peaks from fructose. Two of these peaks do not resolve completely. This presents no difficulty, however, since all three of the fructose peaks are completely separated from all other sugars listed. The fructose can be quantitated by planimetry or electronic integration.

There is essentially no limitation as to what products can be analyzed by the approach described in this paper. For products of high moisture content it may be necessary to take the product close to dryness with a roto-evaporator prior to derivatization.

CONCLUSIONS

The direct trimethylsilylation of food products by methods described in this paper provides a rapid and effective method for separating α -lactose and sucrose. The quantitation of lactose enables calculation of milk solids content in a variety of food products.

KEY WORDS

Gas chromatography, trimethylsilyl, derivatives, lactose, sucrose, food.

REFERENCES

- (1) McInnes, A. G., Ball, D. H., Cooper, F. P., and Bishop, C. T., J. Chromatog. 1:556-557 (1958).
- (2) VandenHeuvel, W. J. A., and Horning, E. C. Biochem. Biophys. Res. Commun. 4:399, (1961).
- (3) Bishop, C.T., in D. Glick, "Methods of Biochemical Analysis", vol. 10, Interscience Publishers, Inc., New York, N. Y. 1962, p. 1.
- (4) Makita, M., and Wells, W. W. Anal. Biochem. 5:523 (1963).
- (5) Sweeley, C. C., Bentley, R., Makita, M., and Wells, W. W., J. Am. Chem. Soc. 85:2497 (1963).
- (6) Rickey, J.M., Rickey, H.G., and Schraer, R. Anal. Biochem. 9:272-280 (1964).
- (7) Sawardeker, J. S., Sloneker, J. H., and Jeanes, A. Anal. Chem. 37:1602-1604 (1965).
- (8) Mason, Blanche S., and Slover, H. T. J. Agr. Food Chem. 19:551-554 (1971).
- (9) Clapperton, J. F., Holliday, A. G., J. Inst. Brewing, 74:164 (1968).
- (10) Slanski, J. M., Mosby, R. J., J. Chrom., 35:94 (1968).
- (11) Cayle, T., Viebrook, F., Schiaffino, J., Cereal Chem., 45:154 (1968).
- (12) Blum, J., Koehler, W. R., J. Gas Chrom., 6:120 (1968).
- (13) Brolost, K. M., Lott, C. E., Cereal Chem., 43:35 (1966).
- (14) Alexander, R. J., Garbutt, J. T., Cereal Chem., 37:303 (1965).
- (15) Ellis, W. C., J. Chrom., 41:325 (1969).
- (16) Kheiri, M.S.A., Birch, G. G., Cereal Chem. 46, 4, 400 (1969).
- (17) Saunders, R. M., Walker, H. G., Cereal Chem. 46, 1, 85 (1969).
- (18) Beadle, J. B., J. Agr. Food Chem. 17, 904 (1969).
- (19) Averill, W., Perkin-Elmer Instrument News, 18, 10 (1967).
- (20) Standards for Grades for the Dry Milk Industry, Bulletin 916, American Dry Milk Institute, Inc.

TABLE 1

Comparison of GLC and ferricyanide analysis of ice cream for total sugars.

	PERCENTAGE	
	GLC	FERRICYANIDE
Glucose	0.94	
Sucrose	12.43	
Lactose	6.40	
Maltose	Trace	
	19.77	19.4

TABLE 2

Percentage Buttermilk solids in Biscuit Mix as calculated from GLC lactose determination.

Biscuit Mix with Added Buttermilk Solids (%)	Percentage Lactose Measured	Calc. Buttermilk Solids (%) (% Lactose/0.518)
1	0.55	1.06
2	1.21	2.34
3	1.60	3.09

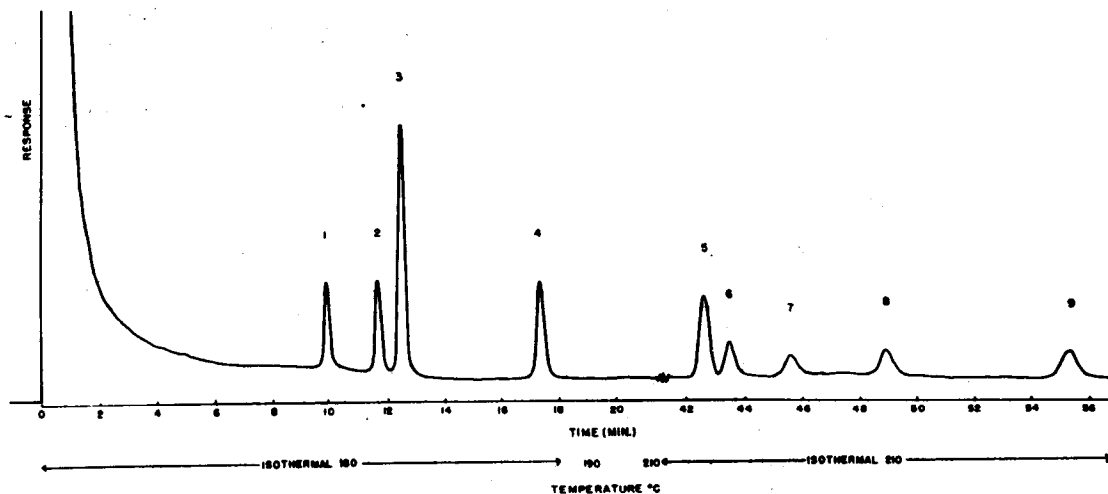


Figure 1
Chromatogram of a Standard Sugar Mixture

1, β -Fructose-TMS, 2, α -Glucose-TMS
3, Sorbitol-TMS, 4, β -Glucose-TMS, 5, Sucrose-TMS,
6, α -Lactose-TMS, 7, α -Maltose-TMS,
8, β -Maltose-TMS, 9, β -Lactose-TMS.

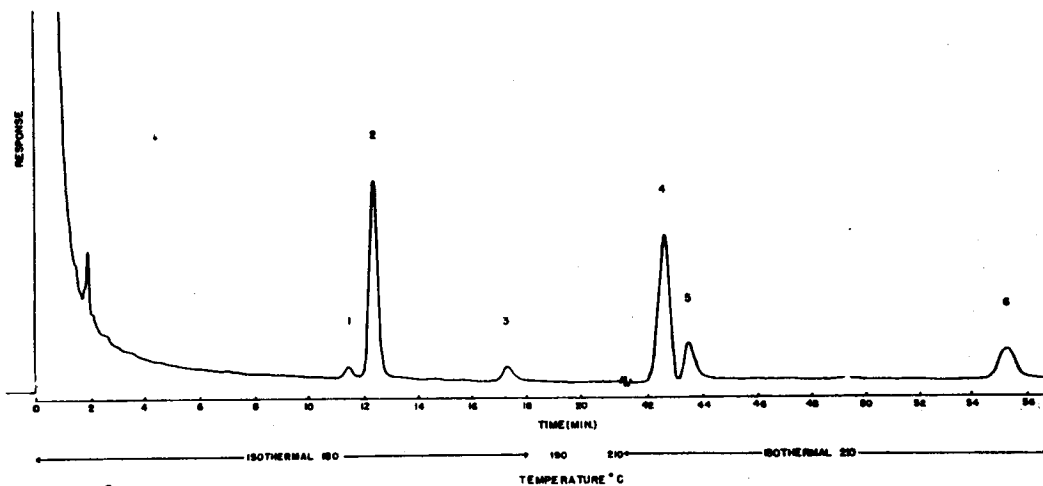


Figure 2
Chromatogram of Sugars in a Commercial Ice Cream.

1, α -Glucose-TMS, 2, Sorbitol-TMS,
3, β -Glucose-TMS, 4, Sucrose-TMS,
5, α -Lactose-TMS, 6, β -Lactose-TMS

Instrumented Color Measurements

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ABSTRACT

The visual assessment of color, although the final arbiter in consumer acceptance of a product, has many problems associated with it.

These include the fact that eyes must be present within a living body to be used; and all the biases, personality differences and associated human frailties present, subject the color judgement to many different interpretations. As well there is the problem of the near impossibility of describing a color in subjective terms.

Instrumental color measurement, when applicable, can overcome many of these problems and produce standardized descriptions of a color which are reproducible and not subject to human error. Such measurements may be carried out on a wide variety of instruments which employ the principles of spectrophotometry, tristimulus colorimetry, or visual colorimetry. However, measurements recorded by such means should be examined carefully in order to insure that such data or some reduction of it actually correlates with a subjective judgement. Instrumental measurements, although an invaluable aid, must correlate with what the eye is seeing in order to be used properly.

INTRODUCTION

The specification of a color is simply a means of locating a point in three dimensional space. From this it follows that colorimetry or instrumental color measurement is simply a means whereby the location of such a point may be described in terms of numerical values. A common question which arises in this area of instrumentation is; why do we have to use instruments and numerical values to describe a color? After all everyone who has normal color vision can use his or her eyes and make a judgement, why is there a need for instruments?

Unfortunately eyes must be present within a living body in order to make a subjective judgement and all the biases, personality differences and associated human frailties present, subject

the color judgement to many different interpretations. This is one problem associated with a color judgement, but another one which is equally important is the near impossibility of describing a color in subjective terms; what do the terms dark red, musty yellow or lavender pink really mean? This problem may be exemplified by a quote from Robert Louis Stevenson when writing from Samoa in 1892 to a friend in London. "I should rather like to see some patterns of unglossy -- well, I'll be hanged if I can describe this red -- it's not Turkish and it's not Roman and it's not Indian, but it seems to partake of the two last, and yet it can't be either of them because it ought to be able to go with vermillion. Ah, what a tangled web we weave -- anyway send some -- many -- patterns of this exact shade".

Obviously there is a need for some sort of objective measurement which would be a function of what the eye sees yet provide a solution to some of the problems which have been enumerated.

In the field of instrumental color measurement we are much more fortunate than those workers in other sensory areas who are attempting, with some success, to create objective measurements. This is due to the fact that people with normal color vision see each and every color in almost an identical manner. Moreover it is known that with the appropriate combination of red, green, and blue lights shone on a screen an observer can match such colors. This may be done simply, as shown in Figure 1, as first described by Newton (1) and Grassmann (2). The color to be matched is projected onto a screen and in addition, each of three projectors shines a red, green, and blue primary light, respectively, on the other half of the screen. There is a complete overlapping of the three primaries to create one spot whose color is changed by changing the intensity of red, green, or blue light striking the screen. Thus any color may be specified mathematically in terms of the intensities of the three primaries required to create a match. Unfortunately it was found experimentally that all colors cannot be matched by the addition of three primaries, even if these primaries are spectrum colors.

"Superior numbers refer to similarly-numbered references at the end of this paper."

However, this problem can be overcome simply by adding one of the primaries to the test color and matching the combined color by the addition of the other two primaries.

Then, for purposes of describing the test color, the light added to it may be thought of as being subtracted from the other two primaries.

That is, the test color can be described with a positive amount of two primaries and a negative amount of the third.

In order to develop this concept into a workable system of color measurement it was necessary to reach agreement on two separate issues.

The first of these would be to establish the amounts of the three primaries required to match any color throughout the visible spectrum in terms of a representative population of people with normal color vision.

The second would be to arrive at a suitable mathematical system in order to express the coefficients of the primaries in a manner which facilitated their manipulation as color units.

The first problem resolves itself into the definition of a standard observer's response to the visible spectrum which, as stated previously, is similar for all people with normal color vision.

Referring back to the type of equipment illustrated in Figure 1, one may see how such standard observer responses may be obtained. If the test light represented a certain wavelength of the visible spectrum, then an observer with normal color vision could match that color by means of additive or subtractive mixing of the three primaries (R), (G), and (B).

This could be done for each wavelength, and the coefficients or tristimulus values of the primaries could be plotted at various wavelengths to obtain standard observer curves for the visible spectrum. The tristimulus values of the standard observer are represented by the symbols \bar{r} , \bar{g} , and \bar{b} .

By using a random sample of persons with normal color vision, average standard observer curves may be developed which represent the physical stimuli causing each color to be perceived by the brain.

The C.I.E. (Commission Internationale de l'Eclairage), which is the most prestigious body concerned with colorimetry, defined color matching characteristics of the standard observer on this basis in 1931. Several modifications in the definition have occurred since then, but the techniques used remained fundamentally the same.

Having defined the standard observer curves, the C.I.E. had to consider the second problem which was to define appropriate reference stimuli or tristimulus values.

As stated previously, the use of (R), (G), and (B) involves the use of negative quantities. Negative values of color were unlikely to be appreciated by those who were going to use the system, and also these negative values would complicate the mathematical manipulation of color units and tend to hamper the design of photoelectric instruments equipped with mechanical integrators.

For reasons such as this the C.I.E. decided that, although the (R G B) system was appropriate for defining standard responses, another system of reference stimuli should be developed. Thus, X, Y, and Z were chosen by the C.I.E. as imaginary primary lights for the description of colors.

These primaries were selected so that

a) all possible real colors could be "matched" by positive amounts of the primaries;

b) a relatively large range of colors in the yellow-red region could be "matched" with only two primaries (note in Figure 2 that the \bar{z} curve ends in the yellow region and colors beyond this may be matched by \bar{x} and \bar{y} only); and

c) the intensity (luminosity or lightness) of the light needed to make the "match" is specified by the Y primary alone.

Figure 2 shows the standard observer curve in terms of \bar{x} , \bar{y} , \bar{z} . These curves were developed from \bar{g} , \bar{r} , \bar{b} standard observer curves, and it should be emphasized that although \bar{x} , \bar{y} , \bar{z} may be calculated from \bar{g} , \bar{r} , \bar{b} experimental data, the X, Y, Z values cannot be produced experimentally.

Having discussed the rationale for the development of instrumental color measurement it is now appropriate to describe the general types of instruments and their theoretical basis.

As stated previously instrumental measurement may be divided into three parts: 1) spectrophotometry, 2) tristimulus colorimetry, and 3) visual colorimetry.

Consider spectrophotometric measurement of color using the C.I.E. system. An instrumental measurement must measure the physical stimuli in terms of how the eye sees color. The instrument achieves this by using either one of two different arrangements of standard source, test sample and photo-detector.

One is named 0, 45° viewing and is achieved by measuring the light which reflects off the test sample from the source at an angle of 45°. This is known as diffuse reflection and is mainly a function of color.

The other arrangement involves the use of an integrating sphere, which is a hollow metal sphere painted white inside. An integrating sphere collects all the light reflected from the surface of a sample placed against an opening (commonly called a port) in its side. The light collected

for measurement may or may not include specular reflection (mainly a function of gloss) along with the diffuse reflection. Both of these methods are used, but unfortunately they do not give identical results for all types of samples.

Readings are obtained over the visible spectrum and a spectral response curve for the test sample is obtained. This response is symbolized by R .

The monochromatic light used is a C.I.E. standard source that has a defined spectral curve of its own. The energy elicited at every wavelength of this curve is symbolized by E . Thus the total energy received by the phototube is the product RE .

In order to obtain values analogous to what the eye sees, this energy function RE must be multiplied by the functions \bar{x} , \bar{y} , and \bar{z} , of the standard observer curve, respectively. The multiplication may take place at intervals across the visible spectrum, so that at any one wavelength, $X = RE\bar{x}$, $Y = RE\bar{y}$, and $Z = RE\bar{z}$.

If the wavelength interval across the visible spectrum was $d\lambda$ then the color of the object may be specified by integrating between 380 and 750 nm as follows:

$$X = \int_{380}^{750} RE\bar{x}d\lambda$$

$$Y = \int_{380}^{750} RE\bar{y}d\lambda$$

$$Z = \int_{380}^{750} RE\bar{z}d\lambda$$

Figure 3 shows this operation in a diagrammatic manner that might be easier to understand.

The integrals could be calculated manually, but normally instruments have a mechanical integrator which carries out these operations so that a direct read-out of the tristimulus values X , Y , and Z is obtained.

In order to obtain the tristimulus coefficients or chromaticity coordinates, these values are merely expressed as fractions of their total so that

$$x = \frac{X}{X + Y + Z}$$

$$y = \frac{Y}{X + Y + Z}$$

$$z = \frac{Z}{X + Y + Z}$$

The second type of instrumental color measurement is that obtained using a tristimulus colorimeter. In this case the integration procedure using standard observer curves is replaced by using

filters which simulate the \bar{x} , \bar{y} , and \bar{z} curves respectively, as shown in Figure 4.

The third type of instrumental color measurement involves the eye as a photocell. In this sense the method is subjective, but mathematical specifications are still provided by the instrument making the description of the color objective. These instruments are widely used in certain areas and may run the gamut from disc colorimetry to color comparators, to the use of standardized glass filters whose combinations form the units which specify the color to refined spectrometers or colorimeters using the eye as a detector.

There are many different types of instruments based on the preceding principles. As well as different instruments, there are a large number of different color solids. All such solids represent three dimensional color space and are mathematically interconvertible. The choice of a given solid is dependent upon the final use and fit of the data to visual judgements as well as the degree of uniform chromaticity desired.

The C.I.E. system represents one of the most common scales or solids and one method of visualizing this is shown in Fig. 5. Here the spectral colors are plotted on x , y coordinates to obtain the C.I.E. horseshoe shaped spectrum locus. All colors fall within this locus but, in order to obtain a point in three-dimensional space, $\% Y$ (specified as the brightness or luminosity function) is also plotted. This means that any color may be specified in three dimensional space by a statement of x , y , and $\% Y$.

Another simple set of scales is that in which a pure white sample (MgO or $NaSO_4$) is taken to read 100 when viewed through each of the filters. Readings obtained are often called G for green corresponding to C.I.E. tristimulus value Y , R for red, related to the large peak of the X curve, and B for blue related to the Z curve.

In an instrument of this type (three filters), values of X , Y , and Z can be approximated by adding a fraction of the B reading to the R reading in order to compensate for the small peak of the X curve. Then the modified R reading plus the G and B readings may be multiplied by appropriate factors to obtain tristimulus values.

C.I.E. Y for MgO is 100 so that when the G value is set to 100 it becomes equal to Y by definition. Adding a fraction of the B reading to the R reading assumes that the small peak of the X curve is the same shape as the Z curve. This is not strictly correct and therefore some instruments use a fourth filter, R' , to simulate the small peak of the X curve. Tristimulus values are then calculated in the same manner as above.

Another common set of scales developed by Hunter (3) are used on some instruments. These produce results in terms of L , which is a lightness function and simulates Y ; a , which predicts redness if positive and greenness if negative, and

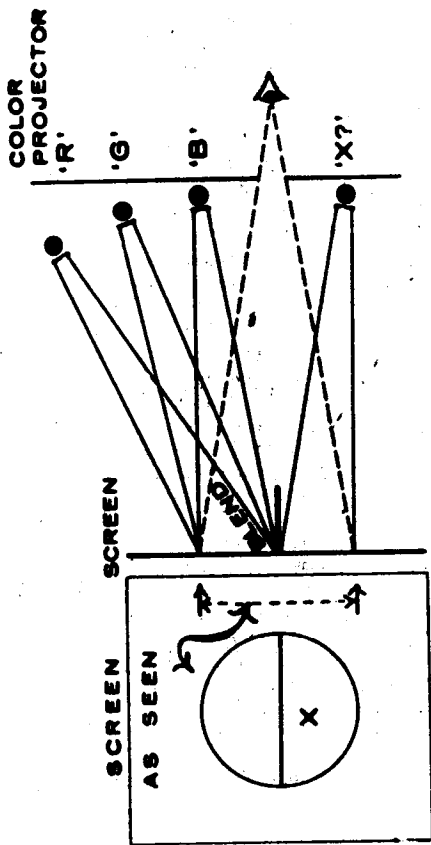


Figure 1. Color matching by the addition of lights

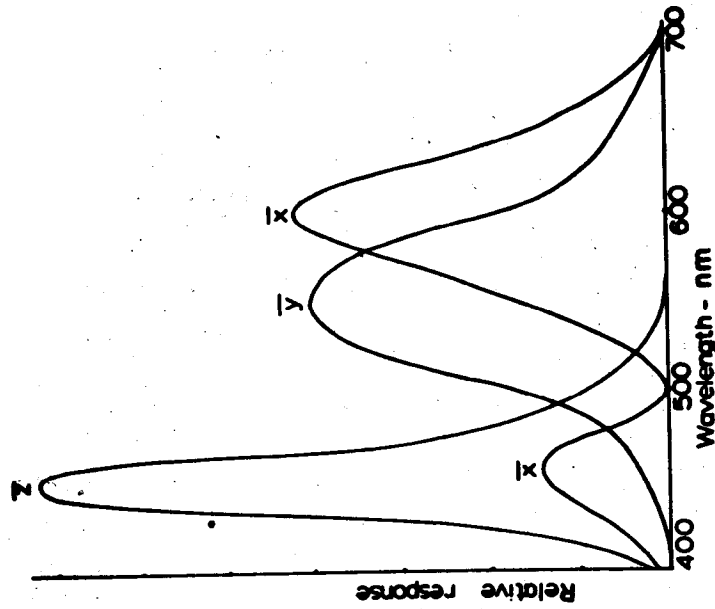


Figure 2. Standard observer curves based on the CIE, XYZ system

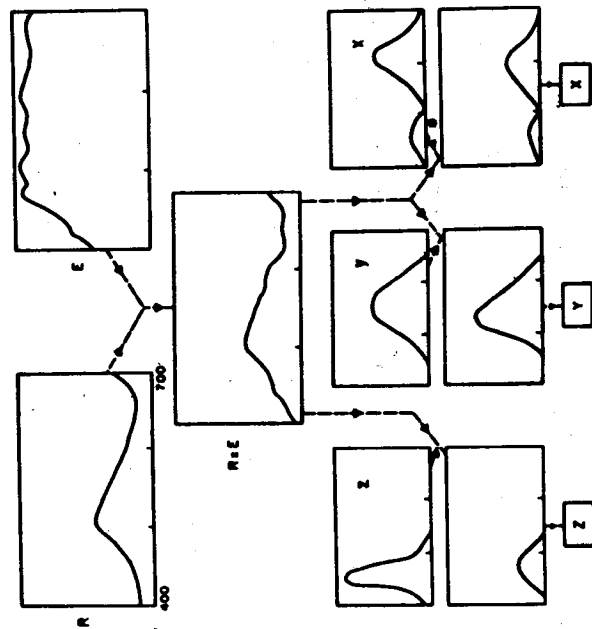


Figure 3. A diagrammatic illustration of the calculation of the X, Y, and Z primaries.

b which predicts yellowness if positive and blueness if negative. A diagrammatic representation of the Hunter solid is shown in Figure 6.

All conversions described above are calculated within the instrument, so that direct read-outs are obtained.

Figure 6 also shows the three functions which make up the Munsell solid. This is perhaps the best known of all the color order systems. It is a system which attempted to create painted colors to represent equal intervals of visual perception of color differences between adjacent samples and also to specify these colors in terms of its three coordinates; hue, value, and chroma.

Hue describes what the average person thinks of when he speaks of color, i.e. red, yellow, green, blue, etc.

Value describes the lightness or darkness of the color.

Chroma describes intensity.

As may be seen in Figure 6, hue is represented on the horizontal circumference of the solid, value is the vertical central axis and chroma is described in units measured outward from the central axis.

There are many other scales or solids, too numerous to mention in this discussion, however a review of this area has been written by Francis and Clydesdale (4).

CONCLUSIONS

There is no question that instruments currently available can measure color as the eye sees it. However, this type of measurement operates with total efficiency only when we are speaking of a white homogeneous solid. Unfortunately there are few food materials which fall in this category.

As pointed out by Clydesdale and Francis (5), the following physical phenomena may occur when light encounters any object.

1. Reflection from the surface.
2. Refraction into the object.
3. Transmission through the object.
4. Diffusion.
5. Absorption within the object.

All of these factors must be considered when measuring the color of a food material, be it solid or liquid, and appropriate steps taken to minimize differences from the ideal.

Another problem encountered with food materials is sample presentation as discussed by Clydesdale and Francis (5). Specialized adoption of instruments must be considered when sampling non-

homogeneous samples such as apples or cranberries or striated samples such as corn on the cob.

However, once the problems are solved the instrument will serve as an objective judge and data may be manipulated to produce meaningful results for research or quality control. For instance since color measurement basically consists of the specification of a point in three-dimensional space, it is logical to assume that the value of each of the three coordinates will change as the color is changed.

Under normal circumstances, however, such as in quality control, it would be advantageous to have only one or two variables, rather than three. If only one or two of the variables are used, there is normally going to be a corresponding decrease in the accuracy of the measurement made. Therefore, it is the operator's choice to decide if the extent of the increase in accuracy justifies the computations involved with the use of three variables.

It should be emphasized that any function of color used for a particular product should first be correlated with a subjective panel of visual judgments. At times there is a tendency to use a function which describes visual judgement for one product on a completely different product. This is a dangerous practice and the literature shows that most single functions which combine the color coordinates are recommended for a single product.

Examples of this may be seen in the function

$$\frac{a_L}{\sqrt{a^2 + b^2}} / L$$

developed by Yeatman et al. (6) used for grading tomato juice and in the recommendation of Francis (7) to use the function $\tan^{-1} a/b$ rather than a/b when measuring the color of apples.

Another type of specification used in color work is the specification of color differences between samples, expressed as a single value.

There are many methods by which this color difference may be specified, but it should be remembered that a color difference score relates the total distance two colors are apart in color space, but does not specify the direction one is in relation to the other. This means that other functions which relate a direction should be used in conjunction with a color difference score.

At times it is convenient to transform one set of tristimulus values to another. Thus, one might specify the degree of greenness or redness by $-a$ or $+a$ respectively rather than specifying three tristimulus values of X, Y, and Z. This is completely legitimate, and there are transformation equations available to do this operation. However, these equations are based on opaque standards, and as Clydesdale and Francis (8) have pointed out, do not always function as well with translucent food

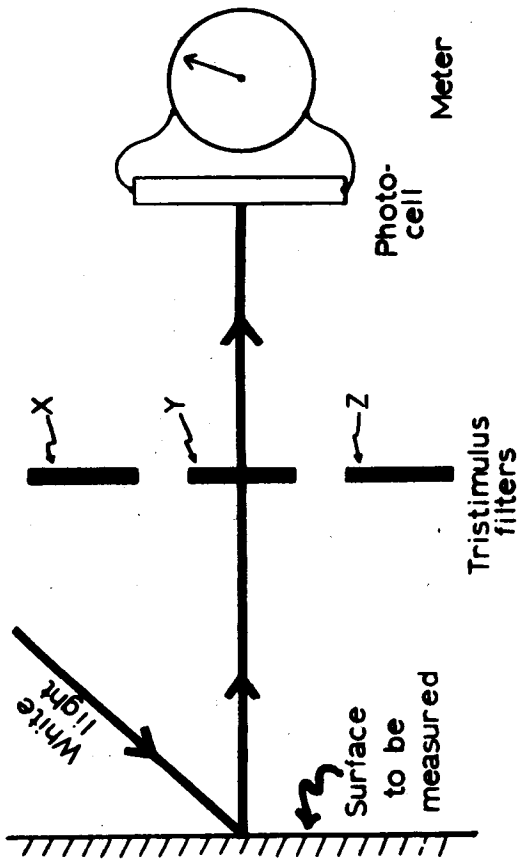


Figure 4. A simple tristimulus colorimeter

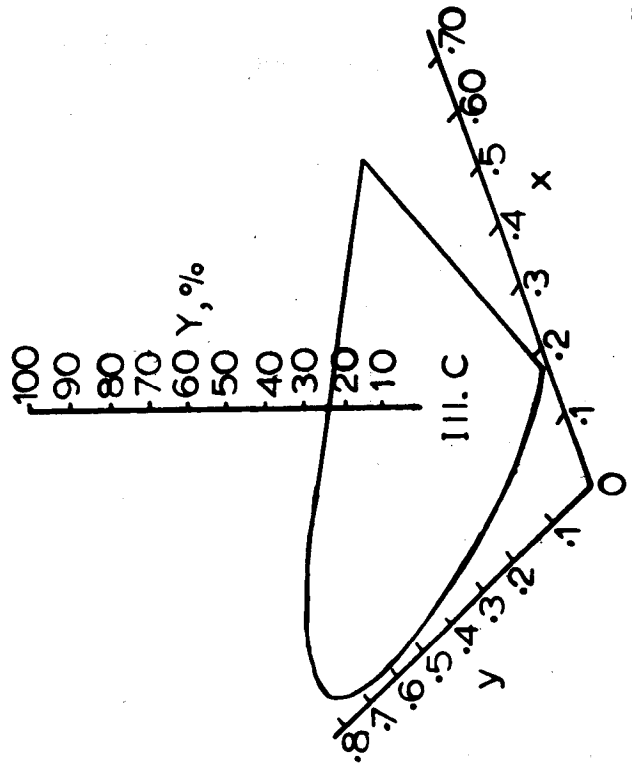


Figure 5. The CIE x-y chromaticity diagram showing percent Y

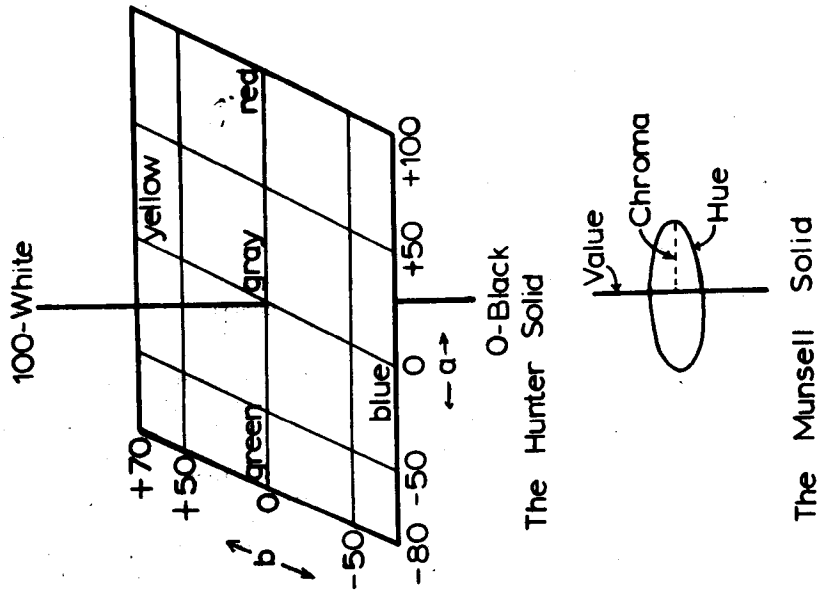


Figure 6. The Hunter and Munsell solids

materials.

Instrumental color measurement is indeed an invaluable aid for the Food Industry. However, as is the case with most technological aids, simplicity does not occur until the problems are totally defined and then resolved.

KEY WORDS

Color, Instrument, Measurement

REFERENCES

- (1) Newton, Sir Isaac. 1730. "Optiks, or a Treatise of the Reflections, Refractions, Inflections and Colours of Light." Reprint based on the 4th Ed., London, 1730. Dover Publications, New York, 1952.
- (2) Grassmann, H. 1853. Zur Theorie der Farbenmischung. Ann. der Physik und Chemie 89, 69. Quoted in Billmeyer, F. W. Jr. and Saltzman, M. 1966. "Principles of Color Technology" p. 31. Interscience Publishers, New York & London.
- (3) Hunter, R. S. 1942. Photoelectric tristimulus colorimetry with three filters. NBS Circular 429, U.S. Govt. Printing Office. Quoted from J. Opt. Soc. Am. 32, 509.
- (4) Francis, F. J. and Clydesdale, F. M. 1969. Color measurement of foods: XIV. Color Scales. Food Product Development, 3, (6), 117.
- (5) Clydesdale, F. M. and Francis, F. J. 1969. Color measurement of foods: XIII. Sample presentation-physical attributes which influence measurement. Food Product Development, 3, (5), 23.
- (6) Yeatman, J. N., Sidwell, A. P. and Norris, K. H. 1960. Derivation of a new formula for computing raw tomato juice color from objective color measurement. Food Technol. 14, 16.
- (7) Francis, F. J. 1952. A method of measuring the skin color of apples. Proc. Am. Soc. Hort. 60, 213.
- (8) Clydesdale, F. M. and Francis, F. J. 1968. Color measurement in foods. 1. Correlation of raw, transformed and reduced data with visual rankings for spinach puree. Food Science 34, 349.

