effective than gossypol in the form of hexane-extracted meats as a precursor of the yellow component, probably because of gossypol inactivation in the diet, as reported by Heywang *et al.* (*6*). Because of interfering pigments, it was impossible to identify gossypol after it was added to the stock diet, but it could be distinguished in the purified diet, as shown in Figure 5, *B*.

Discussion

The results presented here establish a direct relationship between gossypol in the diets of hens and the presence in their fresh egg yolks of a yellow component that can be estimated by quantitative spectrophotometry. This relationship provides the basis for a bioassay of cottonseed meals for available gossypol, and should be particularly valuable in the development of new methods of processing cottonseed.

The role of this component in the discoloration of gossypol eggs during storage is not yet known; studies of discolored eggs show that the distinctive parts of the absorption curve can still be found after 18 months of storage; thus the discoloration is probably not due simply to a breakdown of the yolk component during storage.

Acknowledgment

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MEASUREMENT OF FOOD CHARACTERISTICS

Application of Potentiometric Rotary Viscometer to Measuring Consistency of Food Purees and Pastes

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CONSISTENCY AS APPLIED TO FOOD MATERIALS refers to the subjective impression of "thickness," "body," or resistance to flow. The degree of this property possessed by many food materials—tomato pastes, purees, and catsups, for example—often determines consumer preference and, therefore, the intrinsic market value of the product.

Consistency of food pastes and purees results from the combined contributions of many complex factors. Tomato paste, for example, is basically a two-phase system containing various proportions of a more or less viscous serum and suspended pulp of varying degrees of colloidal suspension and hydration. The enzymatic destruction of pectic substances in the cell walls and middle lamellae of tomato fruit, which may occur during processing (*2*, *3*), results in a marked decrease in hydration of the cellular material, and a corresponding increase in the proportion of liquid serum. For this reason retention of the integrity of pectic substances during processing is one of the most important factors in the production of high-consistency tomato products.

The logical measure of consistency in food pastes and purees would be viscosity. However, in systems such as tomato paste the combined contributions of fiber, liquid serum, and hydrated colloids result in a viscous property which is non-Newtonian and exhibits varying degrees of thixotropy. Consistency is, therefore, difficult to measure in these materials by ordinary viscometric methods.

Rotating cylinders and disks tend to beat paths in such materials, and thixotropy causes liquid films to form at the surface of contact of rotor and medium. Results obtained under these conditions have little meaning. Falling-ball types of measurement are not practical because of the extreme thickness of some pastes and purees, as well as their lack of homogeneity.

Two instruments which have been used with moderate success in determining the consistency of tomato catsups and pastes are the Bostwick Consistometer (*1*), which is frequently used for measuring catsup consistencies, and the Penetrometer, which has been employed largely in this laboratory (*4*) to measure tomato paste and catsup consistencies. The former instrument gives satisfactory results, but is limited to the relatively narrow range of consistencies found in catsups. The Penetrometer has a larger range but is subject to certain disadvantages.

This paper describes the application of a potentiometric viscometer with special rotors (Jacobs potentiometric gel-time viscometer; other types of rotor equipment may be used with equal success) to the measurement of consistencies in food products such as

The consistency of food pastes and purees has been measured with a potentiometric rotary viscometer having two types of rotors—one a straight-sided fork and the other a wire spiral—activated by a synchronous motor through a torsion spring. Displacement of the spring is indicated in degrees of voltage drop, or current flow, which have been calibrated in centipoises against solutions of known viscosity. Results indicate that the instrument and rotors employed are well suited to consistency measurements and largely overcome problems associated with the heterogeneous and thixotropic nature of pastes and purees.

tomato purees, catsups, and pastes. Results obtained indicate applicability over a wide range of consistencies.

Description of Assembly

The two rotors are illustrated in Figure 1. The spiral-shaped rotor is regularly supplied with the instrument employed. The rotor shaped like a tuning fork was selected from a number of experimental models. The nature of the action of these rotors is primarily responsible for the successful application of this assembly to consistency measurements.

The action of the spiral rotor is self-evident from inspection of Figure 1. The rotor consists of a stainless-steel tapered wire helix. Its action is such that any path formed by a given turn of the spiral is continuously closed by the action of other turns of wire. The test medium is submitted to a force tending to "pump" it up the turns of the spiral, and it is mainly the force with which the medium resists this pumping action that forms the basis of the consistency measurement in tomato pastes.

Applicability of the fork-shaped rotor to the measurement of consistency in tomato purees and catsups is largely fortuitous. It happens that, in the consistency range of these products, flow is sufficiently rapid to ensure that the path formed by one leg of the fork essentially closes before the next leg comes around. The upper range of the fork rotor overlaps the lower range of the spiral rotor and it is, therefore, possible to cover a large consistency range without altering other instrument parameters. The total range of viscosities covered by these two rotors is about 300 to 10,000 centipoises.

In the assembly with which the rotors were used they were coupled to a synchronous motor through a torsion spring. A slider on the rotor drive makes moving contact with a potentiometer winding on the motor side of the spring. Electrical connections are made to the potentiometer windings and the slider through a system of brushes and conducting rings built into the potentiometer mounting.

Resistance of the test medium to movement of the rotor causes an angular displacement of the rotor relative to the driving motor and against the force of the torque spring. When the force of the torque spring just balances the resisting force of the medium, the rotor turns at a synchronous velocity with a fixed angular displacement relative to its no-load position in relation to the motor. This displacement is reflected in the position of the slider on the potentiometer winding. When a small voltage is applied across the potentiometer winding, the displacement of the slider may be continuously indicated in terms of a small current or voltage drop by a milliammeter or a voltmeter. This reading is proportional to the angular displacement of the rotor and, therefore, to the viscosity or consistency of the medium under observation.

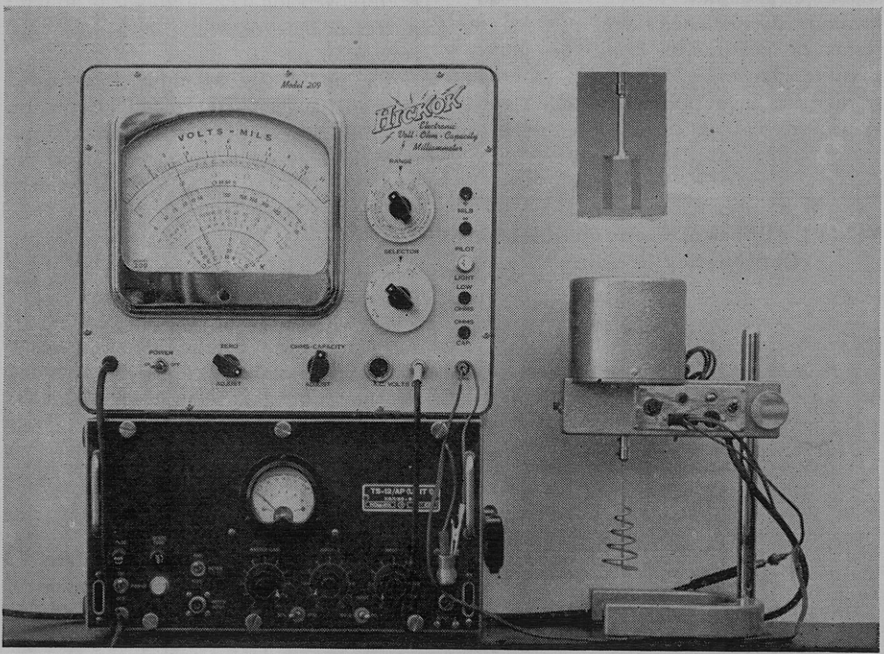
In place of the milliammeter indicator assembly the authors have employed an electronically regulated source of a small constant voltage (a battery would suffice) and a vacuum-tube voltmeter to indicate the voltage drop in the potentiometer winding. This indicating assembly is seen in Figure 1. The high impedance input of the voltmeter draws practically no current, and, therefore, this type of indicator has an inherently more linear response than the milliammeter. Connections to the instrument have been made so that the numerical voltage reading is inversely proportional to consistency.

The rotors were originally designed to measure viscosities and gel times in very thick media which set up into gels on standing. The standard instrument used with these rotors was equipped with a 25 r.p.m. synchronous driving motor. Preliminary studies showed that insufficient torque was developed at this angular velocity for consistency determinations in tomato pastes, purees, and catsups. After testing several possibilities, it was found that the most satisfactory results could be obtained with these products by replacing the 25 r.p.m. motor with one operating at an angular velocity of 150 r.p.m.

Standardizing the Readings

The readings obtained are in arbitary voltages which are dependent on the

Figure 1. Spiral rotor and assembly with fork rotor shown in inset



source of voltage, the potentiometer resistance, and the type of indication used—i.e., voltage or current. Other parameters of the measurement, such as internal resistance of the instrument bearings and the absolute value of the torque spring, may also vary. It is, therefore, desirable to calibrate the arbitrary readings in terms of some reproducible standards of reference. The reference standard chosen was the Hoeppler viscometer centipoise viscosity reading on arbitrary solutions of high viscosity.

For calibration preparations of honey, corn sirup, glycerol, and combinations thereof were employed. Viscosities of these preparations were determined in the falling-ball viscometer, and readings of the same preparations were made in triplicate using the spiral and fork rotors. Determinations were made at 25° C. in both cases. Separate calibration curves for each rotor were then prepared by plotting the averaged voltage readings of the indicator against the log of the viscosities in centipoises determined with the falling-ball instrument. Obviously, if a secondary standard such as the falling-ball instrument is not available, calibration would have to be carried out with standard solutions of known viscosity. Calibration curves for the spiral and fork rotors are shown in Figures 2 and 3, respectively. As may be seen, the curves form approximately straight lines except at the extreme lower limit of the readings, as indicated by the dotted lines. Similar calibration curves have been prepared at 30° C.

The calibration curves may be used as a common basis for reporting and exchanging results, and for recalibration of the readings from time to time. Because of the approximately linear relationship, the determination of two or three points along the linear portion of the curves should be adequate to re-establish calibration. Consistencies are reported in terms of centipoises from the curve. Because, however, the food materials studied are in no sense Newtonian with respect to viscous properties, the term "apparent viscosity" is used in reporting consistency in centipoises. The value measured in food products, although expressed in centipoises by virtue of the calibration method, is in no sense a true viscosity and bears little, if any, useful relationship to true viscous properties.

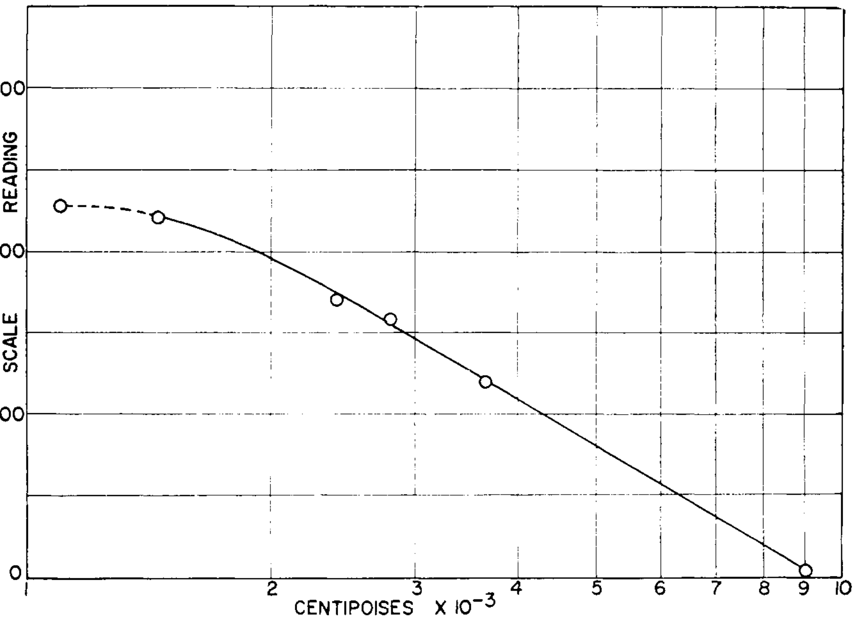


Figure 2. Calibration curve of spiral rotor at 25° C.

Table I. Effect of Container Size on Consistency Readings

Can Designation[a]	Apparent Viscosity, Centipoises Paste, spiral rotor[b]	Apparent Viscosity, Centipoises Catsup, fork rotor[b]
202 × 308	4907	597
202 × 314	4917	597
211 × 400	4907	583
603 × 700	4917	550

[a] Numerical designations indicate diameter and height of can in that order. The first digit of each number represents inches; the next two digits represent sixteenths of an inch.

[b] Average of three determinations.

Application to Tomato Products

Method of Measurement. The sample in question is brought to constant temperature (25° C.) before measurement and transferred in small portions to a 6-ounce (202 × 308) tomato-paste or 6-ounce (202 × 314) citrus-concentrate can with moderate tamping to prevent entrapment of air pockets. In the case of tomato paste, measurements may be made in the original can. The fork rotor is employed for measurements of tomato purees or catsups, while the spiral rotor is used for pastes.

The indicating assembly is adjusted to read between 0.00 and 2.50 volts on the voltmeter scale for maximum and minimum angular displacement of the rotor shaft. The rotor is lowered into the test material, so that the top of the working portion of the rotor is about 0.25 inch below the upper surface of the medium and the rotor is started. The voltmeter scale reading is taken after the rotor has been running for 1 minute. The scale reading obtained is converted to the equivalent centipoise value by means of the calibration curve and the consistency of the sample is then expressed in terms of centipoises of apparent viscosity.

Quadruplicate determinations of consistency have been made on ten samples of tomato paste and ten samples of tomato catsup under the above-mentioned conditions. The average deviation from the mean value found for these determinations was not greater than ±0.05 voltmeter scale unit. This represents only 4% of the available voltmeter scale of 0.00 to 2.50 volts.

Factors Influencing Determinations. Temperature is, of course, one of the most important factors influencing the determinations, and it is essential that all samples be run at the same temperature or that calibration curves be constructed for the temperatures employed.

The depth to which the rotor is immersed in the sample is another important factor when the spiral rotor is employed. When the top of this rotor is too near the surface of the sample, a portion of the sample is pumped above the surface level and forms a standing peak in the center of the container. This results in increased loading of the rotor, and when this condition exists the readings obtained are about 5% higher than otherwise.

With thixotropic materials there is a slow drift of the reading with time. The magnitude of this effect, at least in the case of tomato products, is small in comparison with the 1-minute reading time allowed for short-term equilibrium to be established.

Container size has a smaller effect on the readings than might be expected. As may be seen from Table I, determinations of consistency with the spiral rotor are practically independent of container size. In the case of the fork rotor, a large increase in container size results in less than a 10% decrease in consistency reading. Even this difference is relatively small, considering the uncertainty and lack of precision of the subjective meaning of consistency.

The small effect of container size on readings is largely due to the thickness and thixotropic nature of the materials studied. The motion of the rotors does not usually cause a general rotation of the test medium and, therefore, the "drag" of the medium against the container wall is negligible. Also, in the case of the spiral rotor, nearly all the rotor energy is expended in forcing the medium up through the turns of the spiral, and so there is little tendency to impart angular momentum to the medium.

The relative independence of reading with respect to container size is an important attribute. It means that in routine applications involving tomato paste, large numbers of determinations can be made directly on samples in their original containers. In other cases, a number of sample containers of approximately the same dimensions may be employed for a series of samples, thus avoiding the time-consuming operations involved in the successive transfer of a large number of samples into one or two containers of precisely standard dimensions.

Consistency in Tomato Catsup. Consistency in tomato catsups (as well as tomato purees) is measured with the fork rotor. Measurements of ten randomly selected brands of tomato catsups showed a total range of consistencies varying from 450 to 880 centipoises apparent viscosity. Consistencies of these catsup samples were also measured by the Penetrometer and by measuring the distances samples would flow down an inclined trough in a fixed time. The latter measurement is similar to the principle of the Bostwick and Adams Consistometers, which are often employed commercially to measure consistency in tomato catsups. The order of consistencies assigned to the samples was the same by the viscometric determination as by the measurement of flow. However, consistencies determined by the Penetrometer were not always in agreement with the other two methods.

Table II shows a particularly illustrative case of consistency determinations obtained with the fork rotor and Penetrometer. All four catsup samples were submitted to a panel of eight judges, who were asked to give a subjective estimate of the order of consistencies of the samples. As the table shows, the judges were unanimously in agreement with the order assigned by measuring with the fork rotor.

Table II. Comparison of Fork Rotor and Penetrometer in Determination of Tomato Catsup Consistencies

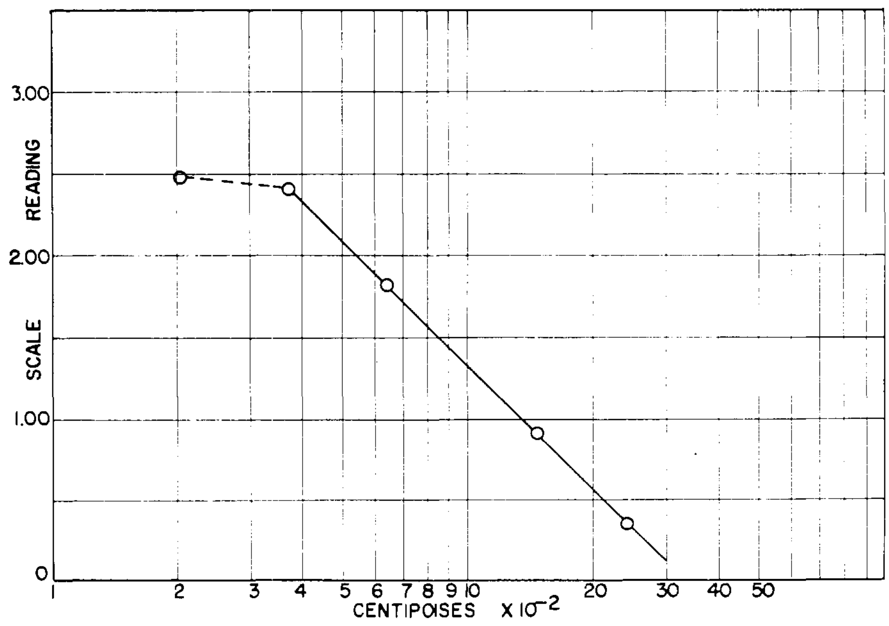
Sample	Apparent Viscosity, Cps.	Penetrometer Reading[a]	Sample Rankings in Order of Decreasing Consistencies: Fork rotor	Penetrometer	Panel of 8 judges
A	880	250	A	A	A
B	830	261	B	C	B
C	610	259	C	B	C
D	510	277	D	D	D

[a] Penetrometer readings are inversely proportional to consistency.

Consistency in Tomato Paste. The spiral rotor is employed to measure consistency in tomato pastes. The consistencies of over 20 samples of tomato paste obtained from different manufacturers have been determined, and found to range from 1650 to 5100 centipoises apparent viscosity. Of the paste samples tested, 50% had consistencies in the relatively narrow range of 3000 to 4000 centipoises apparent viscosity.

The consistencies of the tomato pastes were determined also by the Penetrometer. As in the case of tomato catsup, there was generally good agreement between the determinations, except in the "transition zone" between plastic and fluid properties. Where differences between determinations by the two instruments could be resolved by subjective estimations, the consistency measurements obtained with the spiral rotor were always confirmed. The range of consistencies found occupied 50% of the available scale reading of the voltmeter as compared with only 20% of the available scale of the Penetrometer.

Figure 3. Calibration curve of fork rotor at 25° C.



An example of a research application of the spiral rotor, shown in Table III, furnishes additional presumptive evidence that the determinations do in fact reflect the property of consistency. It is well known that enzymatic destruction of pectic substances during processing (*2, 3*) is a major cause of low consistency in concentrated tomato products. It would be interesting to show that this effect can be reproduced in a commercial tomato paste in which pectic substances are still intact.

A can of commercial tomato paste known to contain relatively undegraded pectic substances was set up in the assembly and the spiral rotor allowed to run until a constant reading was obtained. At this point, 2 ml. of a concentrated preparation of the two pectic enzymes (pectin-methylesterase and pectic acid depolymerase) occurring naturally in tomato fruit (*3*) were injected into the center of the sample with a hypodermic syringe. With the rotor running continuously, consistency readings were made during the time intervals shown in Table III. The first effect of enzyme action is a small increase in consistency. This is the expected effect of the action of pectin-methylesterase which demethylates the pectin present, forming pectic acid, which tends to gel in the presence of polyvalent cations in the tomato paste. Then, as the pectic acid is further degraded by the action of pectic acid depolymerase, the consistency of the paste sample commences a steady decline which is continuing at the termination of the experiment. The loss of consistency was accompanied by an observable syneresis of the hydrated colloids, resulting in an increase in the amount and separation of free serum. The observed rate of consistency loss is relatively slow compared to the effect observed in commercial practice when tomato fruit is macerated in the production of tomato juice products (*2*, *3*). However, the increased free acidity and high concentration of other constituents in tomato paste as compared to a tomato macerate strongly inhibit the activity of the pectic acid depolymerase.

Summary

It is not known that there is any standard of consistency in non-Newtonian media by which it is possible to prove conclusively the adequacy of any type of consistency measurement. Studies on consistency, therefore, must stand on inductive evidence and on the relations between objective determinations and subjective judgments. In this light, these studies are believed to indicate, so far as it is possible, that the viscometer and rotors employed as described measure consistencies in food purees and pastes with a good degree of accuracy and significance.

In the last analysis consistency, as it is related to food products, is a subjective quality. Therefore, no objective instrumental method of measuring consistency, regardless of reproducibility and accuracy, can be regarded as wholly satisfactory unless it can be shown to be correlated with subjective observations. Studies of factors affecting the consistency of tomato pastes, catsups, and purees, some of which are briefly outlined in this paper, indicate a very satisfactory correlation between the instrumental method described and subjective observations. In several cases where the authors have had the opportunity to measure the consistency of tomato paste samples which have also been judged by men long experienced in the manufacture of tomato products, they find that the instrumental determination agrees not only with the relative subjective assignments of consistencies but also with the relative orders of magnitude assigned to the differences.

Table III. Effect of Pectic Enzyme Action on Tomato Paste Consistency

Time, Min.	*Apparent Viscosity, Cps.*
Before Enzyme Addition	
0	3500
5	3400
30	3250
60	3150
75	3150
90	3150
After Enzyme Addition[a]	
0	3150
20	3250
30	3000
45	2900
60	2600
90	2355

[a] 2 ml. of concentrated preparation containing tomato pectin-methylesterase and pectic acid depolymerase.

Although the applications described are limited to tomato products, there is no reason to believe that consistency cannot be determined with equal satisfaction in other food purees and pastes. Preliminary studies have been made of the applicability of the instrument to such diverse materials as mashed potato, pureed baby foods, and fruit purees. The results obtained have been as promising, so far as they go, as the results obtained with tomato products. However, in the case of some products—mashed potato, for example—it is desirable to employ either a driving motor of smaller angular velocity or a smaller spiral rotor to measure the very high consistencies encountered. The range of measurable consistencies may also be extended to lower values by interchanging torque springs or by employing higher angular velocities.

Acknowledgment

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CARBAMATE HERBICIDES

Relative Herbicidal and Growth-Modifying Activity of Several Esters of *N*-(3-Chlorophenyl)-carbamic Acid

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Investigations of the potential utility of the herbicidal and plant growth regulatory activity of derivatives of carbamic acid in the field of selective weed control during the past few years have revealed that two compounds of this class, isopropyl *N*-phenylcarbamate, IPC, and isopropyl *N*-(3-chlorophenyl)-carbamate, CIPC, show great promise as selective herbicides in a variety of crop plants. These compounds are being produced on an increasing commercial scale, especially for the control of annual weedy grasses in a variety of broad-leafed crops.

An earlier paper (*1*) reviewed the history of the development of carbamate herbicides and described the chemical properties and the preliminary results of the screening evaluations of the plant growth regulatory properties of a group of these compounds structurally related to isopropyl *N*-phenylcarbamate, the first of the commercial carbamate herbicides. The present paper describes, in a similar fashion, the chemical properties and the results of screening evaluation of a group of compounds closely related to the second commercial carbamate herbicide, iso-