THE PROBLEM OF ADDED WATER IN MILK, AND ITS DETECTION[1]

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Milk is defined as the "normal lacteal secretion from healthy cattle"; laws have been interpreted to mean that fluid milk for sale to consumers be neither diluted nor concentrated. Milk is an "oil in water" emulsion, and lends itself readily to accidential or intentional dilution with water.

Defective cooling devices which leak water, and equipment from which rinse water has not been completely removed, account for "accidental" entry of water into milk. At farms, after milking machine inflations are dipped into water or sanitizing solution, they must be drained before the vacuum is turned "on." At plants, the water which lodges in pipelines, filler valves, tanks, etc. must be drained completely before operation begins. Furthermore water can not be removed entirely from some presses.

Intentional addition of water to milk, at processing plants, has been prompted by shortage of milk supplies; at farms, by pressure on shippers to increase milk volume on short notice, and more frequently, by shippers' recognition that the price differential for fat above or below the base is much smaller than the return for the base. The lower the fat test and the greater the volume developed by dilution with water, the larger the return for milk delivered to the receiving station.

Example: at $5.00 per 100 lb., for 3.5% milk, with a 4c differential per 0.1% of fat, 100 lb. of 4% milk yields $5.20. Note the yields on addition of varying percentages of water, to lower fat tests, as shown below:

% Fat Mix	Added Water %	Mix Yield Lbs.	Fat Differ. ± $	$ Yield 100 lb.	$ Yield Mix
4.00	0	100	+ .20	$5.20	$5.20
3.90	2.56	102.56	+ .16	5.16	5.29
3.80	5.26	105.26	+ .12	5.12	5.39
3.70	8.11	108.11	+ .08	5.08	5.49
3.60	11.11	111.11	+ .04	5.04	5.60
3.50	14.28	114.28	O	5.00	5.71
3.40	17.64	117.64	− .04	4.96	5.83
3.30	21.21	121.21	− .08	4.92	5.96

A milk's taste is deteriorated, as it is progressively "watered;" its flavor is perceptibly "flat" when 10% of water has been added. Unfortunately, it is impractical to taste milks as they arrive at receiving stations. If this were done, it would be found that frequently, single cans demonstrate as much as 20% of added water. A shipper once explained that an unfilled can floats, in a water-filled cooling tank, and was more readily handled if water was added to permit it to settle!

Serum solids concentrations, as well as fat, are reduced by water additions; the low yields obtained from many batches of cheese, condensed and powdered products, have been traced to the original milks' adulteration by water.

Lactometers will detect milks which have been grossly watered, but are not critical enough to show the presence of less than 10% of added water. The "copper serum" and "sour serum" methods, listed in early editions of Official Methods of Analysis of the AOAC, to detect water in milk, have been abandoned because of their questionable accuracy.

The freezing point range of milk, like that of blood (from which it is formed in the mammary gland) is narrow, excepting only in unusual circumstances (5). When water is added to milk or blood, the freezing point shifts toward "0". That the freezing point method is an accurate one for determining the presence of added water in milk was established almost a century ago. The data developed before 1921, however, is not readily utilizable, since earlier investigators' technics varied widely.

THE CRYOSCOPIC METHOD

The freezing point of pure water is, by definition, 0°C. Ice can, of course, be brought to lower temperatures. Fahrenheit, in 1714, first enumerated the principles underlying freezing point determination. A solution does not freeze as soon as it is cooled to its freezing point, or just below. It remains in a "supercooled" state, while its molecules rearrange themselves into crystallization nuclei. After a sufficient number of these have been formed, the solution freezes spontaneously (2).

The freezing of a super-cooled solution may be induced by vibration, which rapidly rearranges the solution's molecules into the crystal lattice, or by seeding with isomorphic particles, which function similarly. The greater the super-cooling, the more rapid the freezing, but the temperatures of the freshly frozen solution will not truly reflect its freezing point whenever super-cooling is more than slight.

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A lower temperature will be registered if more energy, in the form of refrigeration, is available than is needed for the conversion from liquid to solid state. The super-cooling must be controlled and terminated uniformly to replicate freezing points accurately.

In 1921, Hortvet (*4*) sought to standardize the cryoscopic technic applied to milk to determine the presence and percentage of added water. The equipment and procedure he recommended has been AOAC "official" since the Second Edition (1925) of its "Methods of Analysis." Ether is evaporated to cool alcohol, to super-cool milk, in Hortvet's test; crystallization is initiated by "seeding" with ice. The temperature at this point is observed on a limited range thermometer, calibrated by the operator before use, against solutions prepared from sugar purchased from the U. S. Bureau of Standards.

The ranges of rates of freezing, sample stirring and thermometer tapping, which Hortvet specified, were themselves found to result in variable data. In 1953 Shipe, Dahlberg and Herrington (*7*) assembled a "semi automatic" Hortvet unit in which all these operations were performed mechanically and uniformly, rather than manually and erratically.

The mercury thermometer is still the source of the Hortvet method's weakness. The mercury, chilled in "super-cooling" takes time to react to crystallization temperature; the mass of mercury, large in comparison to sample, affects the end result. The bulb, 65 mm long x 25 mm diameter, "senses" temperatures not at one point, but at many. The mercury, which must be observed under magnification, continually rises and falls. While the "official" instruction (*1*) states the temperature is not to be read "until top of mercury column remains stationary at least one minute," this condition is never reached. Shipe (*6*) showed that the thermometer readings plot into a curve; thus, the reading is taken at a point of least change of slope.

Since the Hortvet scale, by specification, is 30 cm long for 3°C., the smallest graduated interval, 0.01° C., is 1.0 mm long. Estimating to 0.001°C., with the mercury bouncing around, is not as definite as would be desired. With the precalibration and postcalibration required, determining the freezing point of a single sample, even with a semi-automatic unit, is a time consuming chore — and not one leading to satisfaction when qualified, careful technicians, using the same instrument and sample, do not replicate each others' readings.

What was needed to make cryoscopy more critical was a device which could accurately measure minute temperature changes at single points, with the speed of electrical response. In 1939, tiny elements made of fused metal oxides, developed by Bell Telephone Research Laboratories, and called "themisters," did just that when connected into appropriate Wheatstone Bridge circuits. Units made by Fiske Associates, Danvers, Mass., for medical research on osmotic pressure of blood were adapted by the manufacturer for use in determining the percentage of added water in milk (*3*). In all probability, other instruments will ultimately be available from other sources; this discussion below, however, is limited to apparatus made by Fiske Associates, marketed through Advanced Instruments, Inc., Newton Highlands, Mass.

THE THERMISTER CRYOSCOPE

The apparatus and detailed procedure will not be reviewed here, since this information will appear in the forthcoming 11th Edition of Standard Methods for the Examination of Dairy Products, of the American Public Health Association. They are merely indicated below.

The unit is calibrated by means of sugar (or salt) solutions so that its readings are direct and exact without correction to a reproducible accuracy of 0.001°C. — the smallest graduated interval of the control dial. A 2 ml. sample is mechanically stirred while cooled by a thermostatically controlled freezing solution. The progress of cooling is observed on the scale of a sensitive galvanometer. When super-cooling is achieved (a constant below the original dial setting) crystallization is induced by standardized vibration. The temperature rises to the freezing point, which remains constant for fully 90 seconds. At the beginning of this interval, the galvanometer scale is adjusted to "0" by turning the calibrated dial. The entire operation may be observed by all in the vicinity of the instrument. The dial is then read to detail the sample's actual freezing point to the nearest 0.001°C.

The dial is initially set at -0.545°C., on the assumption that the sample is "normal." If the first test establishes that it is not, the dial is left at the reading obtained, to insure that the super-cooling "constant" will be proper for the second trial. The results of the second and third tests will replicate each other to within 0.001°C. on abnormal samples, or on the first and second on normal ones (these latter do not require any more, but will continue to replicate themselves, if retested).

By keeping the test tubes containing the 2 ml. samples in a "precooling" ice-water bath, the time taken per trial will average 2 minutes. It is thus possible to screen hundreds of milk samples per day per unit — with full accuracy, no eye-strain, and corroboration of data by all who are willing to watch.

Whereas it was necessary to take a sedative before

tackling the job of standardizing a Hortvet before performing a single test by that method, a technician will gracefully accept the task of running a hundred samples by the thermister unit — and will report data which is completely reproducible and positive.

Since it is not always possible to obtain "control" samples of milks produced hundreds of miles away, official agencies routinely employing thermister cryoscopes are adhering to the 3% tolerance advocated by AOAC. It has been most impressive, however, that freezing points of policed supplies are soon brought to where they regularly demonstrate completely normal values.

The data from samples taken at some receiving stations (and from some plants) have established that watering, accidental and intentional, has not been as uncommon as some might believe. The particular advantage of the thermister cryoscope is that it takes so little time to perform a test and obtain an accurate figure, that there is no longer any point to just worrying about the possibility of adulteration with water.

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THE LENGTHENING REACH OF THE PUBLIC HEALTH OFFICIAL

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Anyone who looks at the public health field as a whole today can see that the responsibilities of public health people are increasing by leaps and bounds. The developments in industrial chemistry alone have brought on huge new problems in air and water pollution. The atomic revolution, still in its early stages, has already shown that it is going to create unprecedented problems in the handling of atomic wastes and radiation generally. And other technological, industrial and social changes are also bringing a rich crop of problems.

At the same time health officials are adding, as they historically have in the past, to their own concepts of the ground that their departments must cover. As Wesley Gilbertson of the U. S. Public Health Service has pointed out, more than six times as many departments are now concerned with accident prevention as were 15 years ago. And work in the mental health field, control of swimming pools and regulations of homes for chronically ill old people are only a few of the other new activities coming to be regarded as regular health department responsibilities.

But such basic services as food and dairy inspection, water supply and waste disposal, must be strongly maintained. The demand for public health services had its origin in the dangers in these fields that affect large segments of the population — and the guard against these dangers must be kept up.

This combination of pressures, new and old, means a great opportunity for all health officials to grow in stature and importance in both their profession and their community. It also means that the problem of enforcement is considerably more difficult than it used to be. No longer does the public health officer have the time to stand over people to make sure they follow the rules. In all probability he never did have that much time, even when his responsibility covered far fewer areas, but he certainly does not have it now.

This being the case, it should be interesting to see some of the ways in which various health officers and their departments have solved the problem of enforcement. A number have come to our attention in the course of our work for the Public Health Committee of the Paper Cup and Container Institute, and particularly in connection with the gathering of information for the Annual Samuel J. Crumbine Awards.

The most important single thing the Institute has observed is that enforcement is generally easiest when the public health officer has created an environment where the following conditions exist: First, operators want to cooperate, because they themselves have helped to make the rules and know they are