Snowpack Liquid Water Determinations
Using Freezing Calorimetry

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Liquid water in a snowpack has been recognized for some time as a quantifiable variable of hydrologic significance. It is also important in the interpretation of snowpack microwave remote sensing data. One effective method for measuring the liquid water content of snow is freezing calorimetry. This technique is presented from theory through application including formulations for calculating the percent of liquid water in the snowpack. Silicone oil has been used successfully as the freezing agent. Consistent results can be obtained with the method, even when using newly-trained operators. Liquid water content data can be obtained approximately every 15 minutes when using two calorimeters and three operators. The freezing calorimetric approach was found to be accurate in determining the liquid water content of the snow to within ± 1.0-2.0 percent by weight.

Introduction

Liquid water in a snowpack, although recognized for some time as a quantifiable parameter of hydrologic significance, is not a regularly reported snowpack characteristic due to difficulties with its measurement in the field. The amount of liquid water that represents the liquid phase of the snowpack is a parameter that is important in forecasting runoff and predicting the timing of wet slab avalanche release. It is also critically important to the interpretation of snowpack remote sensing data using microwave techniques. Variation in snowpack liquid water

produces changes in the dielectric properties of the snowpack for most of the microwave region which in turn will have an effect on any microwave techniques being used to estimate water equivalent or snow depth.

The liquid water content of a snowpack, sometimes called free water, snow water content, or liquid phase water, includes gravitational water moving downward through the snowpack and capillary water held by surface tension between the individual snow grains.

Various techniques have been used or proposed to measure the liquid water content of a snowpack. Most of these techniques can be broadly categorized as centrifugal, calorimetric, and dielectric and range from laboratory techniques to remote-sensing concepts. Some investigators have tested other methods including Shoda (1952) who used a measurement of volume expansion upon freezing, and Bader (1948) who used the concept of dilution of a solution by the liquid water in the snowpack. Improvement of the solution method for field use has been reported by Morris (1981).

Centrifugal separation of the liquid water from a snow sample was described by Kuroda and Hurukawa (1954) and Carroll (1976). Langham (1974, 1978) subsequently improved the method by isolating the amount of melt that occurs during the centrifuging process. The centrifuge technique has been compared with the calorimetric technique by the National Bureau of Standard (Jones 1979). It was concluded that the centrifuge and freezing calorimeter methods do not measure the same phenomena and that water may be present in the snowpack in a form which is detectable by the freezing calorimeter method but not by the centrifuge method. This is analyzed in more detail by Colbeck (1978).

The dielectric methods for measuring the liquid water content rely on the contrast between the dielectric properties of dry and wet snow. Ambach and Denoth (1974, 1980) have developed capacitance techniques and used them extensively in the field. These dielectric sensors have generally been calibrated with freezing calorimetry. Linlor et al. (1974, 1975) and Linlor and Smith (1974) have used various dielectric techniques, including capacitance and microwave beam attenuation, in the laboratory with some field testing.

Calorimetric methods employ the latent heat of fusion to measure the liquid water in a snow sample from the measured heat balance in a calorimeter. Melting calorimetry using warm water (Yoshida 1940, 1960, 1967) or electrical energy (de Quervain 1946, and Hansen and Jellinek 1957) as the heat source has been tested. These techniques are difficult to utilize in the field and require considerable time for complete operation. Freezing or cold calorimetry using liquid freezing agents has been described by Radok et al. (1960) and used in the field by Leaf (1966). Howell et al. (1976) and Bergman (1978) used the freezing calorimeter with toluene as the freezing agent. Although toluene is satisfactory for freezing the snow liquid water, its toxic properties and relatively high flash point make its use somewhat hazardous.

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Recently freezing calorimetry has been used both in operational-type field work (Fisk 1982) and snow hydrology·research (Denoth 1982a, 1982b). Because freezing calorimetry has been gaining acceptance as a standard method for field measurements of liquid water content and as a method for calibrating other measurement methods, it is presented in more detail here to facilitate utilization by other investigators. Reports on several improvements in the method as well as an evaluation of errors associated with the measurements are included.

Calorimetric Analysis

The freezing or cold calorimeter technique has been selected by investigators primarily because it is relatively inexpensive, it is simple to use, and it is based on known and documented physical phenomena. In addition, it appears that because a small amount of liquid water is frozen in the freezing calorimetric approach as opposed to a large amount of ice melted in the melting calorimetric method, the freezing calorimetric method is more sensitive to variations in liquid water content.

A calorimetric analysis is based on the concept that the heat gained by the system must equal the heat lost. The calorimeter and the freezing agent gain heat, the snow sample and the liquid water lose heat. If the heat gained terms are set equal to the heat lost terms, the following equation results

$$(W_3 - W_2)(t_3 - t_2)C_i + W_{fw}L = [(W_2 - W_1) + E](t_2 - t_1)C_f \qquad (1)$$

where W_{fw} – weight of the liquid water in gm
 $\quad\quad L$ – latent heat of fusion in cal/gm
 $\quad\quad C_f$ – specific heat of freezing agent at $t_1 + t_2/2$ in cal/gm/°C
 $\quad\quad C_i$ – specific heat of ice at $t_2 + t_3/2$ in cal/gm/°C
 $\quad\quad E$ – calorimeter constant in gm
 $\quad\quad W_1$ – tare weight of calorimeter in gm
 $\quad\quad W_2$ – weight of calorimeter and freezing agent in gm
 $\quad\quad W_3$ – weight of calorimeter, freezing agent and snow in gm
 $\quad\quad t_1$ – initial temperature of the freezing agent in °C
 $\quad\quad t_2$ – final temperature of freezing agent and snow mix in °C
 $\quad\quad t_3$ – initial temperature of the snow at sample point in °C

If P is the percent of liquid water in the sample, then:

$$W_{fw} = P(W_3 - W_2) \qquad (2)$$

Substituting in Eq. (1), the expression for the percent of liquid water in the snow sample is

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$$P = \frac{[(W_2 - W_1) + E](t_2 - t_1)C_f}{L(W_3 - W_2)} - \frac{C_i(t_3 - t_2)}{L} \qquad (3)$$

Snow quality, Q_f, as defined by Bruce and Clark (1966) is the percent (by weight) of water in the solid state or (1-P) and can therefore be determined by calorimetric methods.

The type of container which is generally used for the mixing of the freezing agent and the snow sample in cold calorimetry is a vacuum insulated bottle with a temperature probe and a tightly fitted rubber stopper. Experience indicates that a commercial, 1-quart (946 ml), stainless steel, wide-mouth vacuum bottle works well. The temperature probe is used to monitor the changes in temperature that occur in the vacuum bottle during the mixing process. The heat-balance equation requires accurate measurements of temperature changes inside the bottle during the mixing process. Because the vacuum insulated bottle is not a perfect system and some heat is gained by the bottle itself, the heat-balance equations contain a calorimeter constant, E. Each calorimeter bottle will have its own constant that must be determined independently. For convenience in using the heat-balance equations, the calorimeter constant is expressed in terms of equivalent weight of freezing agent. Various methods may be used to determine this constant. The following discussion considers the method developed in the course of this study.

Calorimeter Constant Determination

The calorimeter constant is determined by a basic heat-balance. When a warm fluid is mixed with a cold fluid in a calorimeter bottle, the heat which is lost by the warm fluid must be equal to the heat gained by the cold fluid and the bottle itself. The heat-balance equation is

$$\underbrace{(W_w)\left(\frac{C_w + C_m}{2}\right)(T_w - T_m)}_{\text{Heat lost by warm fluid}} = \underbrace{(W_c + E)\left(\frac{C_c + C_m}{2}\right)(T_m - T_c)}_{\text{Heat gained by cold fluid + bottle}} \qquad (4)$$

where
W_w – Weight of warm fluid
W_c – Weight of cold fluid
E – Calorimeter constant expressed in equivalent grams of fluid
T_w – Initital temperature of warm fluid before mixing
T_c – Initital temperature of cold fluid before mixing
T_m – Final temperature of warm-cold fluid mix
C_w – Specific heat of fluid at temperature of warm fluid
C_c – Specific heat of fluid at temperature of cold fluid
C_m – Specific heat of fluid at final temperature of mix

TARE WT OF BOTTLE — 1040.0 g
TARE WT - COLD SILICONE - 1371.7 g W_c - 331.7 g
TARE WT - COLD + WARM SILICONE - 1478.4 g W_w - 106.7 g

T_w - 7.6°C

$$(W_w)\left(\frac{C_w + C_m}{2}\right)(T_w - T_m) = (W_c + E)\left(\frac{C_c + C_m}{2}\right)(T_m - T_c)$$

$(106.7\ g)(.4373)(37.6°) = (331.7\ g - E)(.4328)(9.7°)$

$E = 86.2\ g$

TIME	TEMP
0	-45.5
1	-44.2
1.5	-43.6
2	-43.1
2.5	-42.8
3	-42.5
3.5	-42.3
4	-42.0
4.5	-41.8
5	-41.5
5.5	-41.3
6	-41.0
6.5	-40.8
7	-40.4
7.5	-40.2
8	-39.9
8.5	
9	-29.7
9.5	-29.8
10	-29.6
10.5	-29.5
11	-29.4
11.5	-29.2
12	-29.1
12.5	-29.0
13	-28.8
13.5	-28.7
14	-28.6

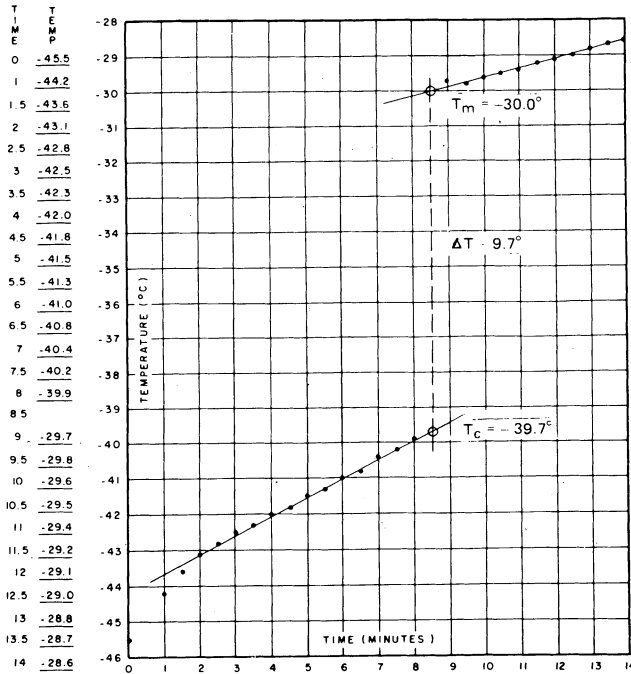Graph labels: $T_m = -30.0°$, $\Delta T = 9.7°$, $T_c = -39.7°$, TEMPERATURE (°C), TIME (MINUTES)

Fig. 1. Typical calorimeter constant calculation worksheet.

The freezing agent used in calibrating the calorimeter in the laboratory was silicone oil. The silicone oil fluid weights (W_w, W_c) can be measured directly and the specific heats of the fluid (C_w, C_c, C_m) can be determined from charts once the temperatures have been measured. Temperature readings are made approximately every 30 seconds prior to and following mixing of the two fluids. The temperature values (T_c, T_m) are determined by standard calorimeter techniques which involve extrapolation of temperature curves. The temperature of the warm fluid (T_w) is measured before mixing. Fig. 1 presents a typical calorimeter constant calculation worksheet which illustrates the procedure. The range of fluid temperatures used in the calibrations should approximate expected field conditions as closely as is practical in the laboratory. When the calibration of a calorimeter bottle is performed under laboratory conditions, the accuracy of the measuring

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equipment and the technique and experience of the calibrator will determine the precision and repeatability of the results. To reduce errors in measurements it is recommended that the average of 10 to 12 calibrations be used to determine the calorimeter constant.

Freezing Calorimeter Equipment and Materials

Certain basic equipment is required for utilizing the freezing calorimeter method described in this paper. First is the calorimeter, which for this study was a wide-mouth vacuum bottle made of stainless steel. Scales capable of weighing up to 2,000 gm and accurate to the nearest tenth of a gm are required for determining the various weights. Finally, 30 cm long stainless-steel thermocouple probes accurate to 0.2°C (used with a multi-channel digital readout) are needed for measuring the temperature changes occurring in the calorimeter. In addition to these basic requirements, several ancillary equipment items are recommended. They are a cold chest for storing the freezing agent, thermal containers for transporting the snow samples, timing devices, and miscellaneous tools including shovels, trowels, and spoons. As a result, the system is not easily portable in the field. Fig. 2 shows the calorimeter with the temperature probe. Fig. 3 depicts the typical equipment needed for field operations set up in the back of a 4-wheel drive support vehicle.

Cold calorimetry requires the use of a freezing agent to freeze the liquid water in the snow sample. It is recognized that a small portion of the capillary water may not be frozen, but the resulting error is considered to be very small. Ideally, a freezing agent should have a low viscosity (down to about −60°C) and a high flash point, be easily obtained, and be non-toxic. Also, it is desirable that the freezing agent leaves no residue or film in the calorimeters. The initial freezing agent used was a chemical grade toluene and subsequently a good industrial grade toluene was found to work equally well. Toluene, although working well as a freezing agent, has two major drawbacks. First, it is toxic, and second, it has a flash point low enough to create a potential fire and explosion hazard, if handled carelessly.

Because of these safety problems, a very light silicon oil (General Electric SF-96-5*) was tested for use as a freezing agent. The silicone oil is more expensive and requires more careful cleaning of equipment than does toluene, but because of significantly improved safety features, it is considered a superior freezing agent. Part of the increased expense of the silicon oil can be offset by the fact that much of it is reclaimable by first letting the ice-silicon mixture warm so that all the ice melts; the water can then be removed and the silicon oil filtered for reuse. Only a small portion of the silicon oil is lost in this procedure.

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* The use of a trade name in no way implies endorsement. The specific name is given so that the reader may investigate its properties.

Fig. 2. Calorimeter bottle with temperature probe and rubber stopper.
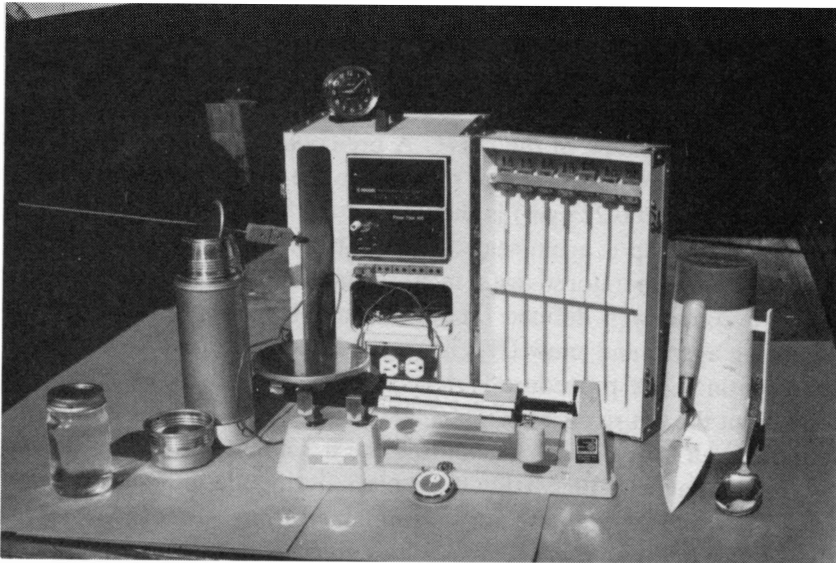

Fig. 3. Freezing calorimeter equipment including calorimeter bottle, digital readout for temperature probes, thermal sample container, scales, trowel, spoon, probes, clock, stopwatch, and bottle of freezing agent.

Field Test Procedure

Two sites in Colorado were used for free water determinations (Jones and Howell 1979). They were located in the southern portion of the Fraser Valley and just south of Steamboat Springs in the Yampa Valley. These locations were used as part of a microwave remote sensing field experiment program. The purpose of these calorimeter tests were to:

1) Evaluate consistency of results using newly-trained calorimeter operators
2) Identify modifications of techniques for improving field operations
3) Determine diurnal patterns of free water at the Colorado sites.

The freezing agent was stored in 1-pint (473 ml) glass bottles in an insulated ice chest containing dry ice. The temperature of the freezing agent was maintained in the range of $-40°C$ to $-50°C$. It is important to maintain this range of temperature, particularly during periods of high air temperatures and high liquid water contents in the snowpack, so that the resultant temperature, t_2, will be less than $0°C$. After the calorimeter bottle has been cooled for approximately 20 minutes (the average time per run) using 1-2 pints (473-946 ml) of freezing agent, the bottle is emptied, wiped dry, and a tare weight (W_1) is taken. One pint (473 ml) of freezing agent is then poured into the bottle and the cap is sealed. At this point the total weight of the bottle and freezing agent (W_2) is obtained and recorded. The temperature of the freezing agent is monitored and recorded every 30 seconds while the bottle is shaken. After the first 3-5 minutes of shaking, the slope of the temperature curve (the heat exchange rate of the calorimeter) becomes fairly uniform. The temperature monitoring is continued for 8 minutes in order to get a good definition of the slope of the curve. A pre-cooled thermos bottle is used to collect and store the snow sample and return it to the field measurement site. The snow (approximately 200-225 gm) is then added to the cold freezing agent in the calorimeter and the cap is again sealed. The bottle is then shaken to thoroughly mix the contents and monitored again in 30-second intervals until the temperature curve again becomes well defined (about 4-5 minutes). The total weight of the bottle, freezing agent, and snow (W_3) is measured and recorded to complete one run of the routine. The procedure is dependent on a good record of time being kept throughout the entire process. The initial temperature curve is obtained from observations taken every 30 seconds during the 0-8 minute interval. The snow is added between minutes 8-10. Mixing takes place during the interval from minutes 10-14, and a second temperature curve is obtained from 30-second observations made during this interval. Both temperature curves (0 to 8 minutes and 10 to 14 minutes) are linearly extrapolated to the 9-minute point when the transfer of heat during the mix is assumed to occur as shown in Fig. 4 for a typical measurement at the Fraser Valley site. At this point the temperatures t_1 and t_2 are determined from
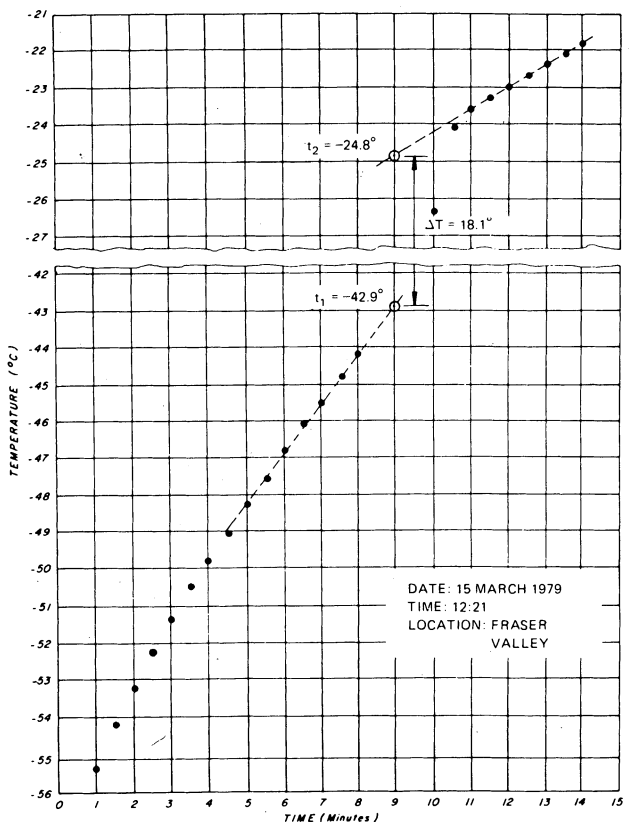
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Fig. 4. Graphical determination of t_1 and t_2 at the Fraser Valley, Colorado site at 12:21 hrs on 15 March 1979.

the two curves. Care must be taken to insure that temperature measurements are taken for a sufficiently long time after adding the snow sample to establish a linear relationship.

Operating the cold calorimeter apparatus under severe winter conditions can be cumbersome and may lead to difficulties in weighing samples and measuring temperatures. Scales and temperature systems are checked for accuracy prior to field operations to minimize basic calibration errors. Field operations are normally conducted with some type of simple shelter to minimize wind and precipitation problems. Despite shelters and precautions, measurement errors are still possible and care should be taken.

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Discussion

During tests at both experiment sites, it was found that various operators could be field trained in freezing calorimetry within several hours. After training, many calorimeter runs spread over several days were conducted. Fig. 5 shows the results of the liquid water content determinations by operator for the Fraser Valley site. Careful analysis of the data generated indicated that there were no consistent differences between operators after training.

The field tests were monitored as a basis for recommending changes or reinforcing the importance of specific procedures for improving field performance. Wind continued to be a problem, and the scales had to be sheltered for accurate measurements. When a road is nearby the measurements can be made in the protection of a vehicle. In remote, heavy snow accumulation areas, a snow pit can be used as an adequate shelter. In doing the tests it was found that three people are definitely needed to run two calorimeters. Because each calorimetric determination takes approximately 20 to 25 minutes, a dual calorimeter system is fairly dependable for readings every 15 minutes. Operators must be inspired and dedicated to obtaining good data, because the continuous running of liquid water determinations quickly becomes tedious. Otherwise, the procedures outlined earlier were found to be very suitable for field operations.

Analysis of liquid water data from a cold spring snowpack at both sites tends to produce a "sawtooth" pattern rather than a smooth diurnal curve. To minimize system errors which might also cause such a pattern, operators were changed from site to site as were calorimeters. This pattern can be visualized by looking at the data from Fraser Valley in Fig. 5. These observations illustrate the fact that the liquid water content in the upper snow layer can vary considerably within a very short time and distance as suggested by Jones (1979). For remote sensing purposes, however, on a meso- or macro-scale, these variations, because of their statistical nature of occurrence, may present a relatively uniform liquid water signature.

It is difficult to precisely estimate the errors associated with this freezing calorimetry technique. Radok et al. (1960) concluded that the freezing calorimeter approach was accurate to ± 1.0 percent of free water by weight based on a detailed error analysis. Following many field experiments, Leaf (1966) estimated that the method yielded results accurate to ± 1.0 percent by weight or about ± 0.5 percent by volume. An error analysis by Jones (1979) concluded that the estimated uncertainty using the method was ± 1.0-2.0 percent by weight. In a series of tests by Denoth (1982b), the liquid water content was measured by a freezing calorimeter with an absolute accuracy of ± 0.5 percent by volume. In the study reported on in this paper, the errors were estimated to be ± 1.0-2.0 percent by weight as a result of running the freezing calorimeter on extremely cold snow in which one would expect the liquid water content to approach zero. An error

Fig. 5. Graphical presentation of air temperature and liquid water content data at the Fraser Valley site, 13-15 March 1979.

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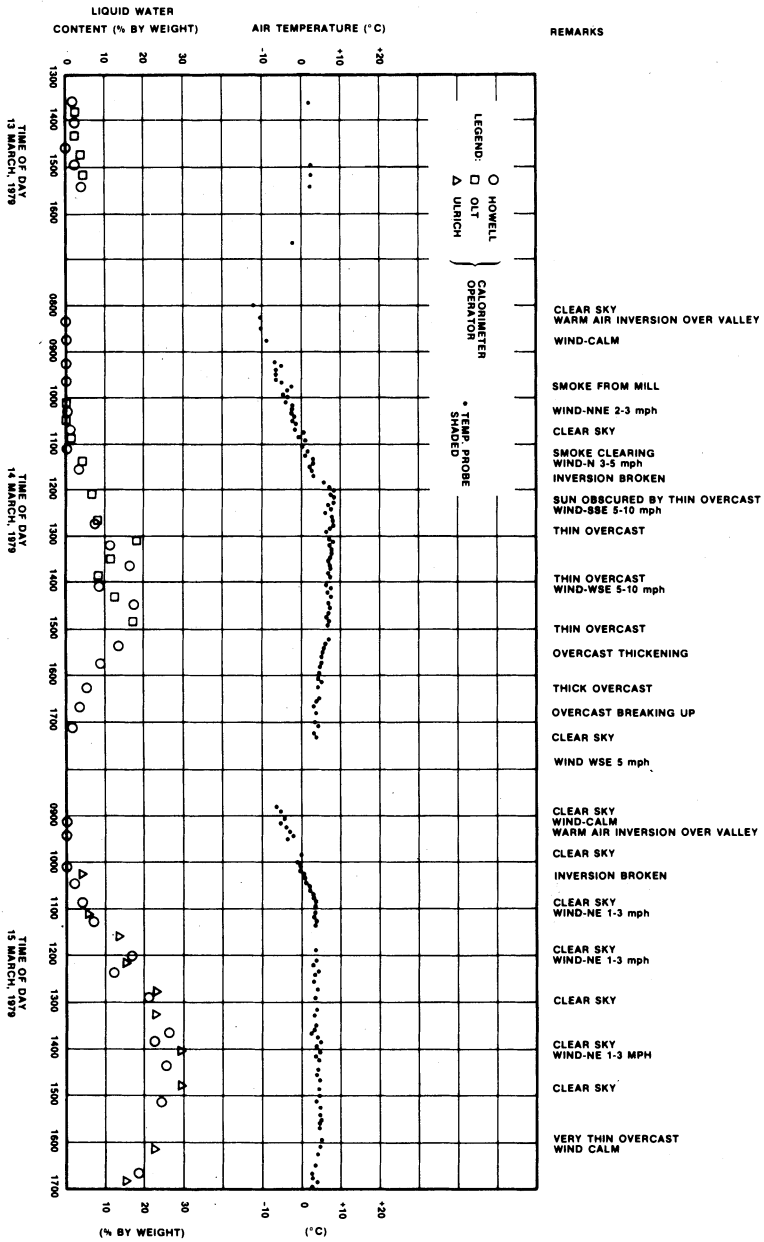
analysis for this study indicated that, in order to minimize error, special care must be taken in the measurement of, first, weight and, second, temperature.

Conclusions

Calorimetric methods employ the latent heat of fusion to measure the liquid water in a snow sample from the measured heat balance in a calorimeter. Because a small amount of liquid water is frozen in the freezing calorimetric approach as opposed to a large amount of ice melted in the melting calorimetric method, the freezing method is more sensitive to variations in liquid water content. Formulations are represented for using the freezing calorimetry approach for calculation of the liquid water content of snow as a percent by weight.

The equipment and procedures currently being utilized in freezing calorimetric determinations of snow liquid water content are relatively simple and easy to use. Light silicon oil has proven to be an effective freezing agent with improved safety features over the commonly used toluene freezing agent. The use of precise digital thermometers in the field operation allows rapid and accurate determinations of solution temperatures. Care must be taken when measuring weights of the calorimeter bottle, freezing agent, and snow sample in the field. Shielding scales from the wind is a necessity.

Training of new operators can be accomplished easily, and no consistent differences in liquid water determinations were found between operators. Each calorimetric determination takes about 25 minutes so that with continuous operation of two calorimeters, liquid water readings can be obtained about every 15 minutes. To accomplish this, three operators should be employed. This frequency of measurement is appropriate for coordinated remote sensing observations during the snowmelt season.

In field tests with the freezing calorimeter technique on a cold spring snowpack, a "sawtooth" pattern of liquid water content was observed instead of a smooth diurnal curve. These observations support the idea that the liquid water content in the upper snow layer can vary considerably within a very short time and space.

Analysis of error associated with freezing calorimetry by other investigators indicated that the approach was accurate to within ± 1.0-2.0 percent by weight. In this study the errors were estimated to be at the same level. To minimize errors, special care must be taken during determination of weights and temperatures.

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