Thermo-physical and Nutritional Changes of Dehulled Yellow Peas During Infrared Processing (Micronization)

By

Sharon Leanne Wray

A Thesis Submitted to the Faculty of Graduate Studies
in Partial Fulfillment of the Requirements
for the Degree of

Master of Science

Department of Biosystems Engineering
University of Manitoba
Winnipeg, Manitoba

THE UNIVERSITY OF MANITOBA

FACULTY OF GRADUATE STUDIES

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ABSTRACT

Infrared processing, or micronization, is an innovative cooking technique that has many benefits and advantages. Micronization is an energy efficient method that converts almost all of the input power into usable radiation. Infrared radiation has the capability for localized heating of a product which results in quick and uniform cooking. During the heating process, the product undergoes physical changes which increase its nutritional value in addition to enhancing its flavor. Micronization has the potential to increase the marketability of hard-to-cook pulse crops by decreasing their required cooking time and creating an instant product.

Experiments were performed with a stationary infrared micronizer to determine the optimum heat intensity required to process dehulled yellow peas in terms of short processing time and promoting high nutrition. A 23.2 kW/m^2 heat intensity at the kernel surface required 60 s of processing time to reach 140°C while 16.8 kW/m^2 required 80 s. Both of these heat treatments increased dry matter digestibility of the peas by 63.0%; increased the amount of available digestible protein by 7%; and required 93% less enthalpy to gelatinize the remaining starch. A moveable multi-lamp micronizer was designed and fabricated based on these results. Although the heat intensity at the pea surface was 23.0 kW/m^2, the maximum average processing temperature was only 105°C. The temperatures inside the kernels were, however, maintained above 95°C for 45 to 130 s depending on their position relative to the lamp, i.e. the length of time the kernels were maintained above 95°C increased with distance from the infrared lamp's initial starting point. To that end, infrared processing is an effective means of processing peas for increased digestibility and decreased cooking time.

ACKNOWLEDGMENTS

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1. INTRODUCTION

Infrared (IR) processing, or micronization, is a relatively new and innovative cooking technique that has proven to be very beneficial. Infrared transfers heat by radiation, which eliminates many industrial heating problems as well as creates a more efficient process than conventional conduction or convection systems (Research Inc. 1994). For example, infrared radiation saves time, money, energy, and space in the cooking and drying industries in addition to many supplemental benefits in the animal production industry.

Micronization transfers heat by infrared radiation directly to a food product for drying or processing purposes. The infrared radiation causes intense molecular action in pulse crops, promoting both uniform internal heating and starch gelatinization which enhances digestibility (Reynolds 1990; Audet et al. 1992). Because of these unique features, the agri-food industry has increased its use of micronization. Previously, micronization was used only as a drying mechanism for various products such as paper and sugar, but now the agri-food industry is applying it to the processing of human food and animal feed.

The concern with micronization is the absence of standards for processing, most important of which is the radiation intensity. The radiation intensity determines the distance between the heat source and the product and the length of processing time. To date, raw feed is processed on a trial and error basis. Usually, a sample of approximately 100 kg is run through the micronizer prior to processing the bulk feed, and the temperature of the resulting micronized product is determined. Too low of a temperature indicates incomplete processing in terms of structure breakdown and the product would, therefore, require a longer processing time. Similarly, too high of a temperature results in over-processing, or burning of the product, and the treatment time should thus be shortened. This method of production is inefficient to maintain a high quality product, and waste of the product, energy, and time are incurred. Also, it is not feasible for the small producer who may process as little as 1 t of material annually.

Manitoba-grown dehulled, dried, yellow peas were selected for this research. North America produces over 160,000 ha of dry peas annually and Canadian production is still expanding (Slinkard & Blain 1998). In 1989, Canada exported 99% of its production of dried peas to at least 30 countries (Anon. 1989, 1994). Peas provide excellent nutritional value as they are high in protein, fibre, and complex carbohydrates, and they are increasingly being researched to replace ingredients currently used in animal feed.

2. OBJECTIVES

The objective of this research was to determine the thermo-physical and nutritional changes of dehulled yellow peas during the micronization process. These factors will contribute to determining the intensity and duration of exposure required for achieving a value-added product. Specifically, the focus of the research was to:

i. determine the optimum energy required for processing;

ii. determine the radiation intensity at the pea surface to optimize the distance between the micronizer and the product to ensure high energy efficiency and high quality;

iii. ensure the nutritional components of the material were enhanced by the process; and

iv. design a scale-model IR micronizer suitable for the home or small company environment.

3. LITERATURE REVIEW

3.1 INTRODUCTION

Today's technology is advancing at a tremendous rate as companies compete to improve productivity and product quality in today's fast-paced world. Part of this process is the adaptation of a previous known technology to new uses. One successful adaptation has been the use of IR processing, also known as micronization, by companies in the food and animal feed industry. This European technique has proven to be very beneficial as it eliminates many industrial heating problems and is a more efficient process than conventional conduction or convection systems (Research Inc. 1994). Infrared processing transfers heat by radiation. Previously, infrared heating has been used in the paper, sugar, and other industries as an efficient drying mechanism, and presently it is being implemented into agri-food processing plants to lower operation and energy expenses, to increase the nutritional value of food, to instantize food products, and to even enhance their flavor.

3.2 INFRARED PROCESSING

The word 'processing' indicates improving, preserving, or changing the product toward a desired end (Nelson 1962). 'Radiation' is defined as the emission and propagation of energy through space or through a material medium in the form of waves (Nelson 1962). Results show, as will later be discussed, that the product is improved in various ways during micronization due to the action of infrared

wavelengths (Reynolds 1990; Audet et al. 1992); hence, the term 'infrared processing' is applicable.

Conventional heating methods of conduction and convection heat an object by physical contact of the heating source and the object or by heating the surrounding air (Abe and Tabassum 1997). Infrared radiation differs because the infrared is radiated by a hot body, and this radiation, in turn, heats the absorbing material (Nelson 1962). Radiation occurs from the rotation and vibration that the molecules of the radiating body experience (Nelson 1962) and the total energy radiated from the source is proportional to the fourth power of the absolute temperature (Nelson 1962). Infrared radiation is directly absorbed by the object, thus eliminating some inefficiencies due to convection.

3.2.1 Early Considerations - Shuman and Staley (1950) suggest three considerations before starting infrared treatment on biological products, and even with today's technology these considerations are still relevant. First is the law of light absorption. "Each hypothetical layer of a given (biological) material absorbs the same percentage of infra-red energy impinging on it." That is, the energy reaching the inner layers will successively be reduced due to the absorption in the previous layers. Figure 1 illustrates this phenomena, showing that the lower the temperature of the emitter, the greater the absorption in the surface layers. This process results in a lower penetration of the energy to the interior of the product. Conversely, with the higher temperature from the infrared source there is less absorption by the surface layers but a deeper penetration of the heat.

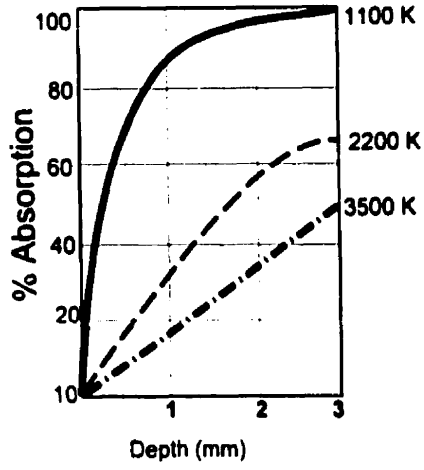


Figure 1 The fundamental law of light absorption (Shuman and Staley 1950)

The second consideration is, "The percentage of absorbed energy varies with the wavelength of the energy (Shuman and Staley 1950)." Wavelengths above 800 nanometers (nm) are beyond the visible light spectrum and will pass through transparent materials such as air. Figure 2 represents a 3 mm depth of water being exposed to infrared radiation. When wavelengths above 1400 nm strike water they are completely absorbed by the water.

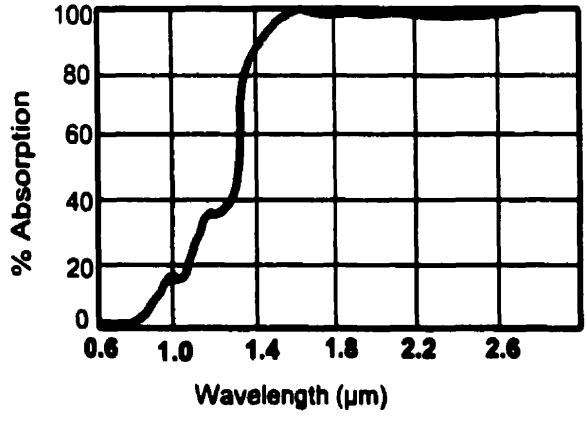


Figure 2 Absorption of infrared radiation (Shuman and Staley 1950)

Lastly (Shuman and Staley 1950) indicated that, "Sources of infrared have wide distributions of energy along the wavelength scale, depending primarily upon the temperature of the source." Each infrared source does have a wavelength of maximum emissive power which represents only a small fraction of the total energy emitted. By increasing the temperature of the emitting object above room temperature it becomes an infrared source which generates a broad band of wavelengths, and the broader the infrared spectrum reaching the micronized product the greater the efficiency of the system. In other words, the infrared source has a broad band of wavelengths that are needed for the greatest efficiency of infrared radiation. Specifically, the infrared wavelengths suitable for micronization are between 1800 and 3400 nm.

3.3 Benefits of Infrared Processing

3.3.1 Quick and uniform cooking - Infrared radiation promotes localized heating, and heating takes place only in the desired area (Research Inc. 1994). This direct heating is possible due to the characteristics of the energy wavelengths associated with infrared. As previously illustrated in Figure 2, wavelengths above 1400 nm are completely absorbed by the micronized object, and the molecules within are excited and vibrate at a frequency of 8.0×10^7 to 1.7×10^8 MHz (Shuman and Staley 1950; Reynolds 1990). This rapid molecular vibration causes uniform internal heating yielding an evenly cooked product in less time than conventional methods (Reynolds 1990). The only possible concern is that micronization is a rapid

process. For example, it may only take 40 to 90 s to micronize pulses. As a result, precise control of the surface temperature of the micronized product is essential otherwise burning or discoloration of the surface of the product could take place.

Nutritious pulse crops such as peas and lentils exhibit a hardshell and hard-to-cook phenomena which result in a slow and variable cooking time. These drawbacks in cooking are major contributing factors to the under-utilization of these products (Iyer et al. 1980; Bhetty 1988; Buckle and Sambudi 1990). Micronization is able to overcome this problem by converting slow cooking legumes into quick-cooking products, and consumption would likely increase.

Cenkowski and Sosulski (1997) demonstrated the effect of micronization on the cooking time of lentils. The results show that both the micronized and control samples of lentils have a significant water uptake during the first 5 min of boiling, increasing by 13% wet basis (wb) moisture content (mc) depending on the sample's initial moisture content. After 15 min of boiling, there was a significant difference in the moisture content. The micronized sample had reached its full moisture uptake at 60% wb mc, however, the control sample did not reach this stage until after about 30 min of boiling (Figure 3). This quick-cooking method occurs because the infrared wavelengths cause the gelatinization of starch within the product, which results in a change of the product's cell structure. This change in structure improves water uptake during cooking thereby decreasing the normal cooking time (Bedard 1996).

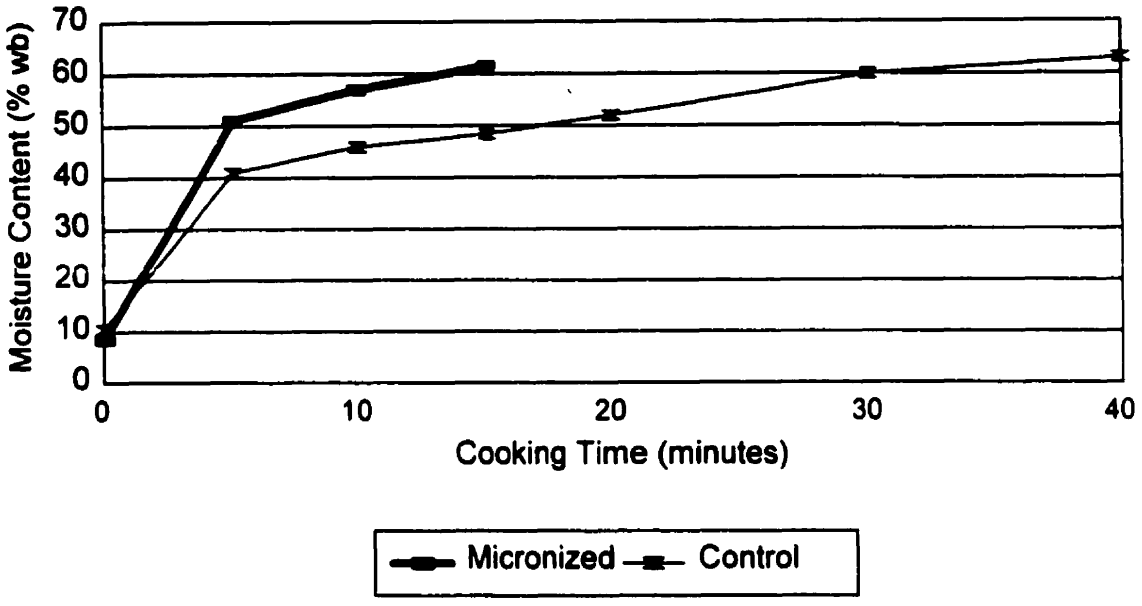


Figure 3 Water uptake by the control and micronized lentils during cooking (Cenkowki and Sosulski 1997)

3.3.2 Digestibility - Infrared processing has also been proven to increase the digestibility of food products (Audet et al. 1992). Many economical and wholesome leguminous seeds are being overlooked in the animal feed industry because of their antinutritional factors such as high starch contents, trypsin and protease inhibitors, flatulence factors, and lectins which adversely affect some people and many young animals (McCormick 1989; Fasina et al. 1997; Owusu-Asiedu 1997). Through the process of micronization, these disadvantages have been overcome (Precision Feeds 1996; Fasina et al. 1997; Owusu-Asiedu 1997). Starch, for example, is difficult for a young animal to digest because each starch granule is surrounded by a tough, elastic wall. However, the rapid action of the inter-molecular friction caused by infrared heat and the increased vapor pressure within the kernels rupture the

granular structures, thereby gelatinizing the starch in the presence of available moisture in the product resulting in improved digestibility (Audet et al. 1992; Precision Feeds 1996). Table 1 refers to five livestock feed grains comparing the digestibility of regular cold-rolled samples to those that have been micronized (Reynolds 1990; Audet et al. 1992). It is evident that micronization greatly increases the digestibility of the product.

Table 1 Comparison of Digestible Starch Levels

Grain	% Digested Cold-Rolled	% Digested Micronized
Barley	32%	98%
Wheat	28%	90%
Corn	43%	74%
Sorghum	48%	72%
Oats	50%	80%

Source: Audet et al. 1992

Furthermore, many enzymes necessary for optimum digestion are insufficient during the first 9 wk of a young pig's life (Figure 4). Following is a description of the functions of these enzymes. Lactase aids in the breaking down of lactose; lipase is essential for breaking down fat; amylases and maltases are enzymes required to digest carbohydrates; and proteases are necessary for the use of proteins from a wide range of sources (Precision Feeds 1996). Micronization allows a producer to match the changing digestive activity of an animal as growth occurs (Precision Feeds 1996). For example, the small intestine (the primary location for digestion

and absorption of nutrients) of a pig changes dramatically during weaning. The capability to absorb critical enzymes such as lactase decreases and irritation by certain proteins increases. By feeding the young pig micronized products, infrared treatment can more efficiently breakdown proteins for better use and maximize nutrient absorption (Cranwell 1995).

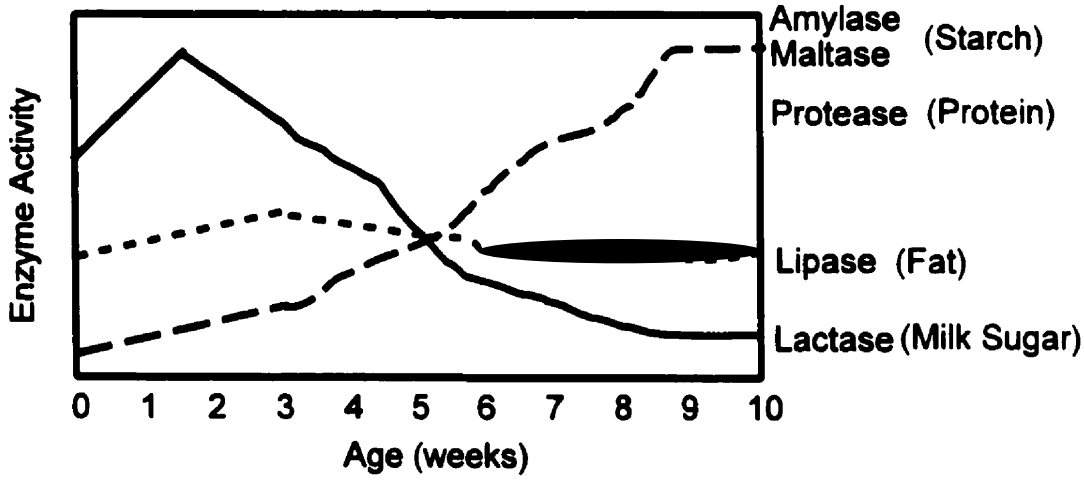


Figure 4 Digestive enzyme activity in the young pig (Cranwell 1995).

Researchers have varying opinions whether the nutritive value of feedstuffs improve with micronization. This could be due to the differences in micronizing techniques, such as the final temperature of the product and the length of processing time. For example, Fernandes et al. (1975) examined the effects of micronization on barley for growing pigs. Barley was micronized to 175°C in 40 s and had 4% more digestible energy over ground grain but the growth rate was not

affected. The crude protein content for the micronized sample was 5% less than the ground barley, and the assumption was the protein content was adversely affected thereby not causing an increased growth rate.

Lawrence (1975) was evaluating the micronization process for preparing cereals for growing pigs. Three different micronized wheat treatments were compared with wheat that was a) hammer-milled through a 4.7 mm screen and b) cold-rolled and then hammer-milled. The micronized treatments were micronized for 23, 37, 76 s until 155, 190, and 220°C were reached. After feeding 60 pigs controlled amounts of feed Lawrence (1975) found the highest micronization temperature resulted in a significant depression in growth rate, and there was no significant difference of efficiency of conversion of dietary dry matter when compared with the other treatments. It was concluded that micronization was not beneficial for wheat fed to growing pigs.

In addition to the increased digestibility of the micronized product, the starch gelatinization which takes place results in a product with a more appealing flavor, that is described as less 'beany' and more 'nutty' (Audet et al. 1992).

McCurdy (1992) performed a study to determine if micronization would decrease the bitter taste in dried peas. Two tonnes of dried peas were processed for 50 s until a temperature of 90°C was reached. The trained tasting panel deemed the micronized peas to be 'more bland' than the control sample.

Kouzeh-Kanani et al. (1981) showed how micronization decreased anti-nutritional factors such as urease and trypsin inhibitors in soybeans while maintaining the available lysin levels. Further, when the soybeans were placed in

an insulated vacuum flask for 25 min after micronization the economical benefits increased because the sample was micronized to a lower temperature, thus needing less time. As a result, the gas consumption decreased.

In these few studies, there is a vast variation in the processing times and temperatures, even given that different products were tested. There was no mention of the actual operating conditions in these papers ie. temperature or intensity of the heat source. It is possible in the first two cases (Fernandes et al. 1975; Lawrence 1975) the materials were over-processed, therefore producing negative results. Also, wheat has a low starch content, so the micronization effect would not be as prominent as a high-starch-content material. Slominski (1997) studied the digestibility of low fibre rapeseed/canola that was moist-heat treated. Figure 5 illustrates the effect different heat treatments had on digestibility. The optimum temperature, in terms of digestibility, was approximately 107-108°C. Treatment temperatures below 105°C and above 110°C resulted in decreased digestibility. Realizing that the comparisons are not exactly parallel, these results could possibly explain the negative conclusions previously achieved; Lawrence (1975) and Fernendes (1975) used relatively high temperatures whereas McCurdy (1992) used a low processing temperature.

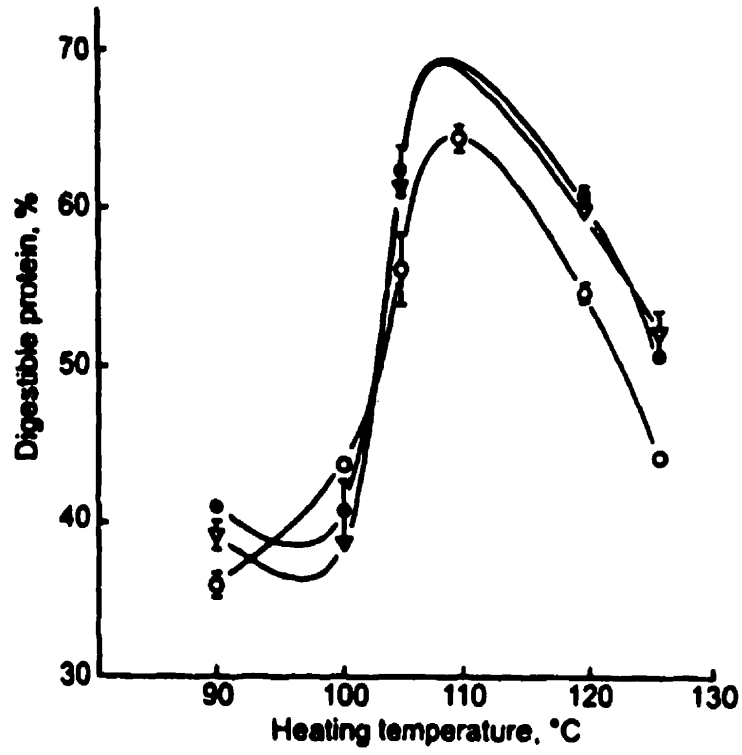


Figure 5 Effects of varying temperatures on digestible protein content in defatted seed samples from three *Brassica* cultivars subjected to moist heat treatment for 20 min (Slominski 1997)

Specifications should be determined to eliminate conflicting results and to produce the best quality product in terms of increased availability of nutritional components and destruction of anti-nutritional factors while maintaining good taste.

3.3.3 Infrared Sources and Effects of Micronization - There are two main generators of choice for infrared processing depending on the wavelength of maximum radiation. The first is a light or short wave radiator which has a temperature of 1500°C and a wavelength of 1300 nm though part of the energy will

still be in the visible light range. Long wave, or dark radiators, operate between 350 to 400°C with a maximum radiation at about 3000 nm.

It is necessary to find the optimum parameters for micronization to be effective. Some of these important parameters include the distance between the infrared source and the product, the temperature of the source, and the length of time for the process to be complete. For example, if the product was to be exposed to high heat for a significant amount of time, it would become dehydrated and somewhat resemble unpopped popcorn (Bedard 1996), whereas the final product should, compared with a non-micronized sample, have a nearly undetectable color change although it may appear to have a slight wrinkling (Bedard 1996).

Fasina et al. (1997) observed that infrared heating caused the surface of leguminous seeds to crack. This cracking occurs because the rapid internal heating increases the vapor pressure (the same explanation for uniform cooking) within the kernel. Consequently, this rise in vapor pressure will cause an expansion within the seeds, thereby increasing the volume of the seeds. This expansion will result in fracturing of the surface material which will cause the product to be less tough than the unprocessed samples.

Bedard (1996) notes that storage for six months had no effect on the cooking time of micronized lentils, as they still could be cooked in half the normal time. More research on the storage of micronized products and the changes they undergo is still required at this time.

3.3.4 Efficiency of Micronization - Many industries can benefit from this new micronization technology as time, energy, and space could be saved. Infrared processing is a clean electric (or natural gas) heat source which never has direct contact with the product. Furthermore, micronization offers efficient use of electrical energy as it converts 88% of input power to infrared radiant energy at the indicated rated voltage by the manufacturer (Research Inc. 1994). Losses occur because the moisture from the biological product is evaporated. This moisture reflects the incoming infrared heat, preventing the heat from reaching the product. Industrial infrared lamps can reach the desired temperature in about a minute yielding fast warmup times at the beginning of production and allowing for the heaters to be switched off if production must stop (Research Inc. 1994). Overall, micronization takes less time than conventional heating methods because the heat is localized and penetrates the surface of the material. This penetration allows for uniform cooking in the interior of the product (Research Inc. 1994; Stupec et al. 1990).

In the paper industry, for example, when the paper is not completely dried, residues are left on the machines causing the machines to breakdown which can result in significant downtime. The efficiency of the operation is increased by using infrared processing as the paper is then uniformly and thoroughly dried (Stupec et al. 1990).

3.4 Future Benefits of the Infrared Process

Infrared heating has been used in various commercial applications such as sugar, grated codfish, potatoes, and fruit and vegetables even before the 1950's (Shuman and Staley 1950). It has also been used for the curing of paints and finishes (Nelson 1962). Today, the paper and animal feed industries are the most prominent industries using the micronization process.

Due to the many benefits of infrared heating, future applications are almost inevitable. Society is becoming an instant and fast-food world. Micronization can aid in the instantizing of historically slow cooking pulse crops. These pulse crops are potential ingredients for soups, sauces, stuffings, salads, and other value-added products (McCormick 1989; Bedard 1996). Already, micronized peas are on store shelves as healthy snack food products.

As well, infrared processed goods could benefit third world and densely populated countries where energy is limited and conservation is a concern. It is an inexpensive process as it requires only the capital cost of the equipment and a low input energy per unit mass of the micronized product (Bedard 1996). Not only will a quicker and more economical product be available but a more nutritious one as well.

4. MATERIALS AND METHODS

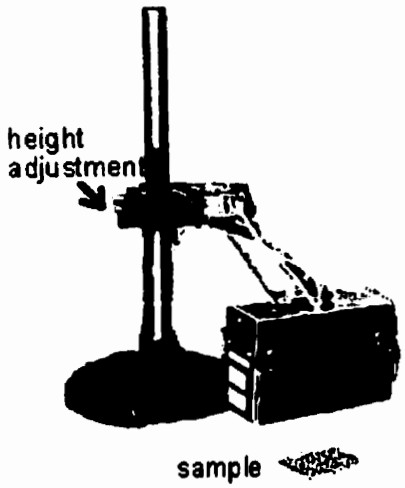
4.1 SAMPLE TYPE

Dehulled yellow peas, grown and harvested in 1998 in Southwestern Manitoba, were provided by Roy Legumex of Landmark, Manitoba in January 1999. The average initial moisture content (mc) was approximately 7±1% wet basis (wb) and initial protein content was approximately 21%.

4.2 EXPERIMENTAL EQUIPMENT

The micronizer components were a Model 4553 High Density Infrared (IR) Pyropanel strip heater (Research Inc, Eden Prairie, MN) mounted on a variable height stand (Figure 6). The heat source consists of four tungsten filament lamps, 500 W and 120 V, with a low resistance at room temperature, and therefore, was used with a Model 5620 Power Controller to eliminate high inrush currents. The heater was a perforated ceramic reflector bonded to a metal back plate with an air plenum and covered electrical connections. Edge reflectors were mounted to restrict radiant energy to a rectangular area and to act as a re-emitter. The reflectors also aided in lessening the bright light emitted from the infrared lamps. The ceramic reflectors have absorption/emission characteristics such that the heater module is maintained at a high surface temperature that continually vaporizes organic compounds (Research Inc. 1998).

a)



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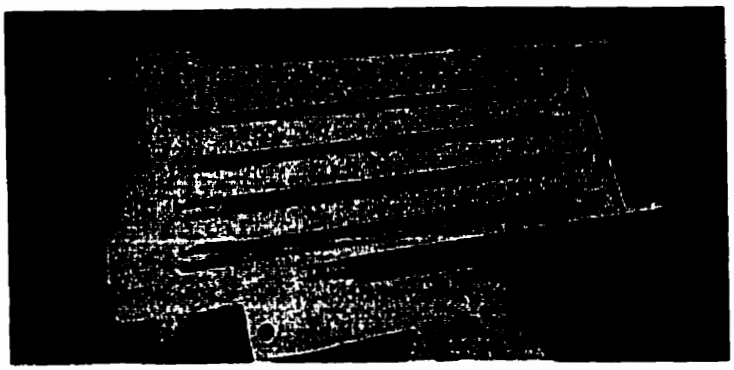

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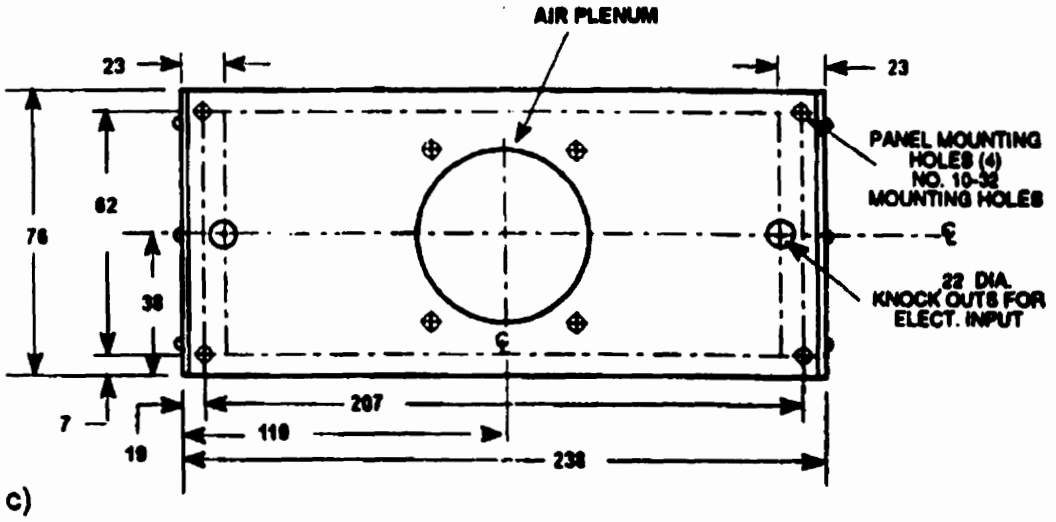


Figure 6 a) The IR multi-lamp stationary micronizing unit
b) The four IR tungsten filament lamps
c) Top view of the Model 4553 High Density IR Pyropanel strip heater (dimensions in mm) (Research Inc 1994)

Ultraviolet rays are not emitted by the IR lamps, but harmful burns could result if contact is made with the surface of the heating module. Dark glasses should always be worn when the lamps are on because of the brilliant light they emit.

4.3 Uniformity of Heat Under the IR Lamp

Before testing samples, the uniformity of the heat given off by the IR lamp was confirmed. Eight porcelain cups, 4 cm top diameter, 1.5 cm bottom diameter, and 3 cm depth, were filled with approximately 10 mL of distilled water and placed equally spaced under the IR lamp (Figure 7). Thermocouples were placed in the middle of each cup and the temperature of the water was recorded with time. Trials were completed for the IR lamp at 6, 8, 10, and 12 cm above the surface of the water.

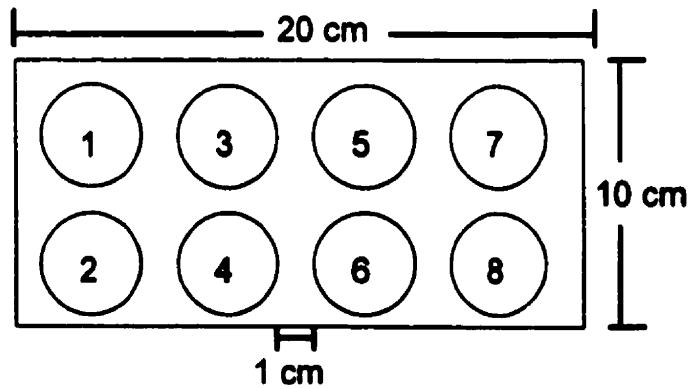


Figure 7 Testing IR lamp uniformity by heating water in porcelain cups under the lamp's lighted area.

4.4 Intensity Determination

The intensity of the IR radiation at the pea surface was determined using the theory provided by Person and Sorenson (1962). Three thermocouples were attached underneath a 0.08 x 0.02 cm copper plate of 2 mm thickness. The plate was lightly coated with soot to ensure equal absorption of heat across the length of the plate. The copper plate was placed at 8, 10, and 12 cm under the infrared lamps until it reached approximately 170°C. Using Equation 1 the intensity of the IR radiation for each height was determined using the same theory as described by Fisher (1996).

$$Q = \frac{\Delta T c_p m}{At} \tag{1}$$

Where:
Q = intensity, kW/m^2
ΔT = change in temperature for duration of test, T_i-T_o °C
c_p = specific heat of copper plate, 0.386 kJ/(kg K)
m = mass of copper plate, 0.0292 kg
A = surface area of copper plate, 0.08 x 0.02 m^2
t = time copper plate was heated, s

4.5 Sample Preparation for Micronization

Dehulled yellow peas were split by shaking them in a glass jar. Their initial moisture content was measured based on the AACC standards (AACC 1983). To obtain a desired moisture content of 26, 28, or 30% wb for micronization, approximately 100 g of peas were placed in a sealable plastic bag, and the appropriate amount of water was added to the peas (Arntfield et al. 1998). The bag

was shaken for equal moisture distribution, flattened to a single layer, placed in darkness at room temperature for 16-18 h, and turned periodically. The tempering procedure was adapted from Arntfield et al. (1998). In our case, a plastic sealable bag was more suitable than a glass jar because the peas were dehulled. When the peas were moisturized in the glass jar they stuck together in large clumps since they do not have seed coats. By placing the seeds into a bag in a single layer, separating was effortless after moisturizing. A sample of the moisturized peas was taken again after 16 h of tempering for accurate moisture content verification. Upon verification, it was shown that the actual moisture content was consistently 2% lower than the desired moisture content. The actual moisture content is referred to throughout the remainder of this paper .

4.6 Temperature Measurement During Micronization

A 20 g sample of split peas was evenly distributed on wire mesh, with the round part of the kernel toward the lamp. Thermocouples attached to an Omega Multiscan/1200 (Stamford, CT) were placed within kernels at three locations under the infrared lamps to determine the temperature change in the kernels and to compare the temperature distribution across the infrared lamp (Figure 8).

The infrared lamp was positioned at 8, 10, and 12 cm above the kernels for different trials in triplicate. These heights were selected based on the preliminary research of Fisher (1996). The infrared lamp was turned on to its maximum intensity until the kernels reached approximately 140 ± 5°C. After micronization, the kernels

were cooled, then sealed in a plastic bag and refrigerated at 5°C for further testing. It was not possible to insert the thermocouples into the kernels moisturized to only 24% wb mc. Thus, the kernels were only moisturized to 26 and 28% mc, henceforth.

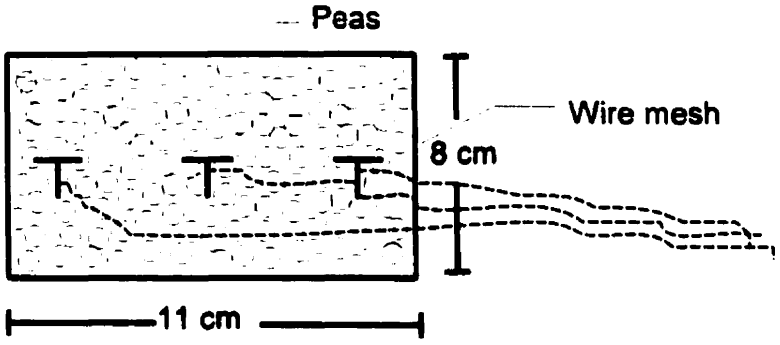


Figure 8 Temperature measurements during micronization with the thermocouples inserted into the kernels. 'T' represents the thermocouple placement (- - - thermocouple wires).

4.7 Mass Measurements

After the peas were split and moisturized to 26 and 28% mc wb, mass measurements were taken using a Sartorius AG Gottingen scale (Edgewood, NY). For mass measurement determination, the wire mesh was placed on an apparatus attached to the scale such that the changing mass of the kernels was recorded without the intense heat from the lamps reaching the scale itself. Two 1 cm thick ceiling tiles with a 1 cm airspace were set on the scale. Tests were performed to ensure the temperature at the scale was not greater than 40°C. The thermocouples were then eliminated to prevent interfering with the mass measurements.

To determine the equilibrium moisture content of the peas, one sample was micronized at a 10 cm lamp height until the surface appeared charred, then the moisture content was determined.

4.8 Determining the Effect of Micronization on the Digestibility of Peas

Differential Scanning Calorimetric tests were performed on micronized and non-micronized peas to ensure the infrared treatment did, in fact, gelatinize the starch found in the peas while maintaining other nutritional components, such as protein.

The temperature and relative humidity (RH) of the room were monitored with a Vaisala Humidity & Temperature Indicator HMI31 (Boston, MA) to ensure constant ambient conditions.

Micronized peas were ground in a Philips coffee grinder for 60 s. The pea flour was left in open air for 24 h to equilibrate with the ambient air (23°C, 35% RH), and then 2-3 g of pea flour was measured for moisture content. The flour was then sifted in a No. 70 (212 μm, 65 mesh) Canadian Standard sieve (The W.S. Tyler Co. of Canada, Ltd.) to ensure the components' availability to gelatinize during the scanning process. Approximately 8.5 mg of the flour was weighed into a steel pan on a Perkin Elmer AD-4 AutoBalance (Norwalk, CT) and water was added to make a 30% solids mixture. After the moisture was added, the capsules were pressed closed and not disturbed.

The Differential Scanning Calorimeter 7 (DSC7) (Perkin Elmer, Norwalk, CT) was calibrated against indium at a melting temperature of 156.6°C and a change in enthalpy (ΔH) of 28.4 J/g (Appendix A). Nitrogen and helium were used as purge gases and an ice water bath was used to cool the unit. Three to five tests were conducted on each micronized pea sample and a reference sample against an empty pan for the scanning range of 30°C to 110°C at a rate of 10°C/min.

The processed samples were not tested in the DSC7 until two weeks after micronization. Thus, further testing was done to determine the level of retrogradation of starch. Three 20 g samples were micronized at a lamp height of 10 cm. This height was chosen because preliminary DSC7 analysis eluded the intensity at 10 cm was optimum in terms of gelatinization of starch and maintaining the protein and lipid contents. The three micronized samples were ground together in a Philips coffee grinder for 60 s. This alleviated any differences in processing among the three identical micronization trials, for example, location of the kernels under the lamp and different lengths of processing time. The DSC7 trials were then conducted immediately, 24 h, and 14 d after micronization to determine the retrogradation of starch.

4.9 *IN VITRO* DIGESTER

Tests were performed in the Department of Animal Science, using the same method as Slominiski et al. (1999), to simulate the digestion process to ensure that the protein content of the micronized material was not detrimentally affected for digestion. Only three samples, in duplicate, could be used due to the length of the

experiments. Therefore, a reference sample (non micronized, non moisturized 7% mc wb) and two micronized samples (26% mc wb) treated at different IR intensities were chosen. This allowed for a comparison of the effect of varying intensities when performed on the same material.

The reference sample and two micronized samples were ground with a 1 mm screen. A 5 g sample and 0.5 g pepsin were weighed and combined in an Erlenmeyer flask with 50 ml of 0.1 M HCl/54 mM NaCl solution. The flasks were covered with paraffin wax paper and incubated for 1 h at 40°C in an environmentally controlled shaker, simulating the stomach. After the incubation period, 2.5 ml of 2.0 M NaOH was added to the mixture to adjust the pH to approximately pH 7.0, and then 250 µl of pancreatin solution was added to represent the chemicals found in the small intestine. The mixture was then transferred, requiring 20 ml of 0.1 M phosphate buffer (pH 7.0) to rinse the flask, into presoaked dialysis tubing with a molecular weight cut off value of 12,000-14,000 (Spectrum, Houston, TX). The dialysis tubing was then sealed by tying the end and allowing for a small air gap in the tube to facilitate continued mixing of the contents. The tubes were submerged in a water bath filled with 0.05 M phosphate buffer pH 7.0, and attached to the digestion/dialysis unit (Figure 9) which simulated the small intestine. The six tubes were left for 6 h of digestion at 40°C rotating in the unit at 20 rpm. After this incubation period, the enzymatic activity was halted and the tubes were subjected to dialysis for 72 h by replacing the buffer with distilled ice water to cool the system to 2°C. The dialysis water was changed twice a day to maintain a constant temperature. Upon completion of dialysis, the contents were transferred into plastic

sealable bags, washing the excess substance with distilled water, and frozen overnight. Once frozen, holes were poked into the plastic bags with a syringe and then freeze-dried for four days. The remaining dry matter was weighed and the contents were then analyzed in duplicate for crude protein using the Kjeldahl method (N x 6.25). Digestible protein content was calculated by taking the difference between the total protein in the undigested reference sample and the protein retained in the dialysis tubing.

The detailed descriptions of the preparation of the solutions for the digestibility tests are given in Appendix B.

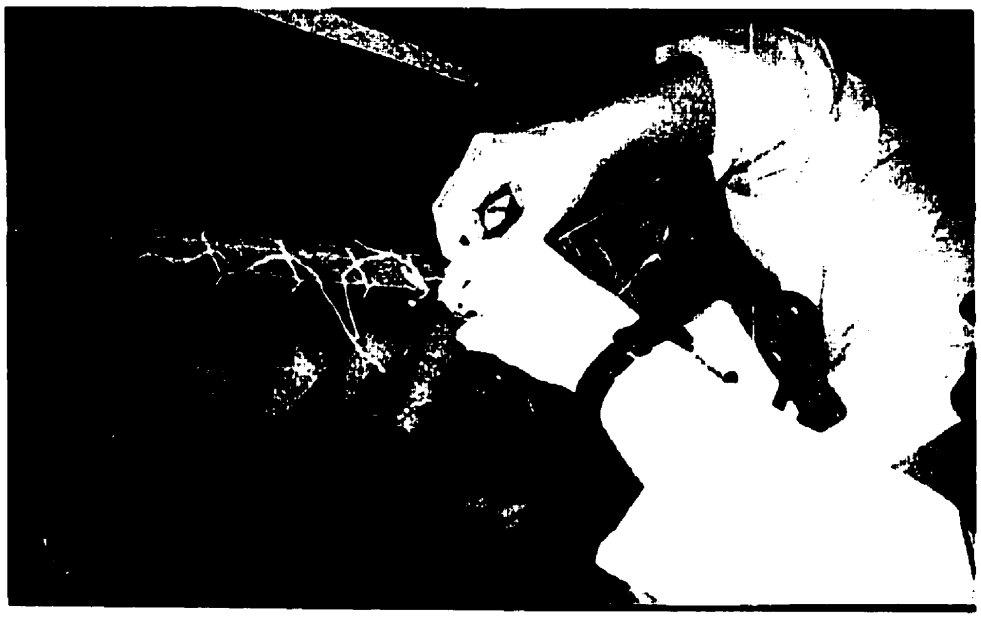


Figure 9 Digestion/Dialysis Unit

5. Results and Discussion

5.1 Heat Emitted from the IR Lamps

5.1.1 Configuration Factor - The amount of energy from the IR heat source actually hitting the peas, expressed in terms of a configuration factor ($F_{1\text{-}2}$), was determined using the theory in Siegel and Howell (1992). A perfect system would have $F_{1\text{-}2}$ = 1.0, or 100% of the energy emitted would hit the pea surface, and be absorbed. The configuration factor is shown in Figure 10 as a function of time and was determined for identical, directly-opposed rectangles using the following formula, which is solved in Appendix C:

$$F_{1-2}=\frac{2}{\pi XY}\left\{\ln\left[\frac{(1-X^2)(1+Y^{2)}}{1+X^2+Y^2}\right]^{1/2}+X\sqrt{1+Y^2}\tan^{-1}\frac{X}{\sqrt{1+Y^2}}+Y\sqrt{1+X^2}\tan^{-1}\frac{Y}{\sqrt{1+X^2}}-\tan^{-1}X-Y\right\} \quad (2)$$

where:

$$X=\frac{a}{c}\ \text{and}\ Y=\frac{b}{c} \quad (3)$$

and:
a = width of objects, 7.6 cm
b = length of objects, 18 cm
c = height between objects, varies from 0.25 cm to 30 cm

Moving the IR lamp source to a height of 6.0 cm above the material yields an $F_{1\text{-}2}$ of 0.51, indicating that 51% of the energy emitted by the source is reaching the material. The $F_{1\text{-}2}$ is 0.38, 0.29, and 0.22 for lamp heights of 8, 10, and 12 cm, respectively.

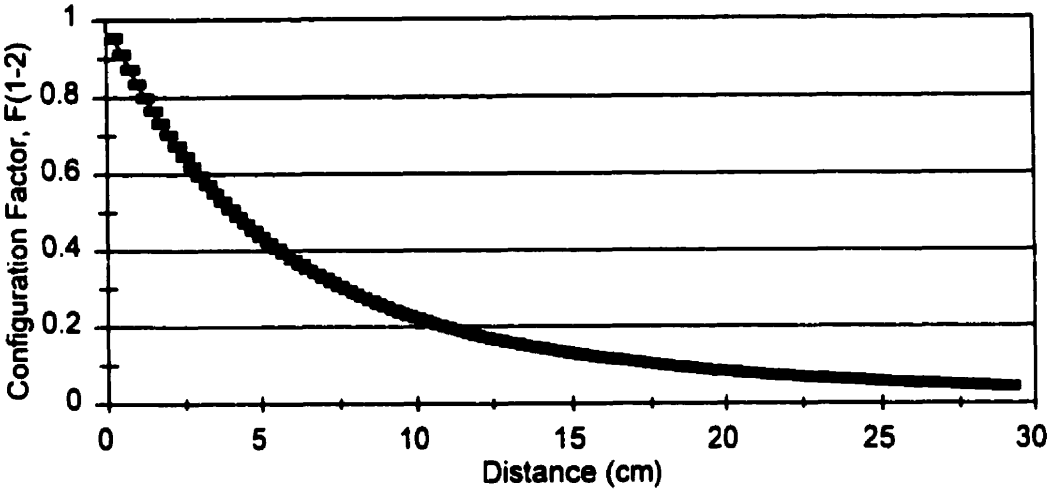


Figure 10 Configuration factor, F_{1-2}, for directly opposed rectangles as a function of distance from the heat source to the kernels.

5.1.2 IR Intensity Determination - Table 2 indicates the intensity of the IR lamps at the pea surface for the various test heights. The intensity was calculated based on Equation 1.

Table 2 **Intensity at Pea Surface at Various IR Lamp Heights**

Height of Lamp (cm)	Intensity (kW/m^2)	Standard Deviation (kW/m^2)
8	23.2	0.6
10	16.8	0.1
12	12.3	1.2

The standard deviation for the 12 cm height is high, perhaps because a different temperature range was used for the third trial. The initial temperature of the copper plate was 111.3°C and the final temperature was 190.6°C, about a 70°C higher range than the other four trials.

Lowering the lamp by 2 cm from 12 cm to 10 cm resulted in an increase of heat intensity by 37% and lowering the lamps again from 10 cm to 8 cm resulted in an additional heat intensity increase of 38%. The difference between 8 cm and 12 cm was 89%. This indicates that the intensity at the pea surface increases exponentially, confirming the configuration factor theory previously presented. The remainder of this paper will refer to the experiments in terms of intensity.

5.1.3 IR Lamp Uniformity - As expected, there was a variation in heat supplied across the lamp. Appendix D provides the temperature histories of the water in porcelain cups as it was heated with the IR heat source. The temperature histories are illustrated by the temperature difference from the initial temperature, since the initial temperatures varied. Table 3 indicates the maximum temperature reached and the standard deviation for each position number under the lamp, for each test height. The position numbers correspond to Figure 7, and are arranged from the position that received the highest intensity to the position that received the lowest.

Table 3 Maximum Temperature and Standard Deviation for Each Position Under the IR Heat Source, Arranged in Descending Order of Intensity

6 cm			8 cm			10 cm			12 cm+	
#	Max ΔT	Max St. Dev	#	Max ΔT	Max St. Dev	#	Max ΔT	Max St. Dev	#	Max ΔT
3	85.8	15.5	4	84.5	11.5	3	87.2	16.1	3†	83.8
4+	84.6	–	6	83.5	20.3	4+	84.4	–	4	83.5
5	81.2	9.1	3	82.8	6.2	5	81.6	1.9	6	83.1
2	74.6	3.8	5	82.7	7.4	2	76.4	4.3	5	82.5
6	73.4	14.2	2	82.4	4.5	6†	72.7	12.3	2	77.8
8	69.9	9.0	8	77.6	3.8	7	76.1	11.5	8†	75.8
7	69.4	11.5	1	75.2	2.6	1	72.1	3.0	1	77.1
1	68.2	3.4	7	59.9	5.2	8	71.2	9.0	7	73.3

† Lower maximum temperature, though overall higher temperature throughout.
+ Only one test used for analysis.

The middle of the heated area (numbers 3-6) received a greater rate of heating compared with the outer positions (numbers 1,2,7,8). That is, 87.5% of the time position numbers 3-6 had the highest four temperatures, and these four temperatures were within 2.0°C, 0.7°C, 2.3°C, and 0.5°C, respectively for 12.3, 16.8, and 23.2 kW/m^2. The outer positions were approximately 5-20°C lower than the middle area. With the exception of position number 7, the temperature distribution varied by 9°C at 23.2 kW/m^2. Position number 7 is 18°C lower than the cup placed beside it (number 8) which indicates an experimental error. The 16.8 and 12.3 kW/m^2 trials each varied by 16 and 10°C, respectively. This overall

difference in heat uniformity should be taken into consideration for the movable micronizer design.

The 6 cm height was eliminated after the uniformity tests for safety reasons despite having a theoretical configuration factor of 0.51. The white reflective paper underneath the porcelain cups started to burn and the surrounding tabletop became hot. In addition, the uniformity experiments did not produce extraordinary results, varying about 17°C from the center of the unit to the edge of the unit.

5.1.4 Temperature Change of Peas During Micronization - The kernels were heated until approximately 140°C, however, visual and audio inspection were also factors. For example, if the kernels were burning, then the process was halted. Also, at some point during micronization, the kernels would snap as the water was being released or evaporating from the kernels. When this noise was slowed down it appeared to indicate that the peas were processed – much like a similar phenomenon during microwaving of popcorn.

Figure 11 shows a representative temperature history of pea kernels for six treatments. All of the temperature histories are shown in Appendix E.

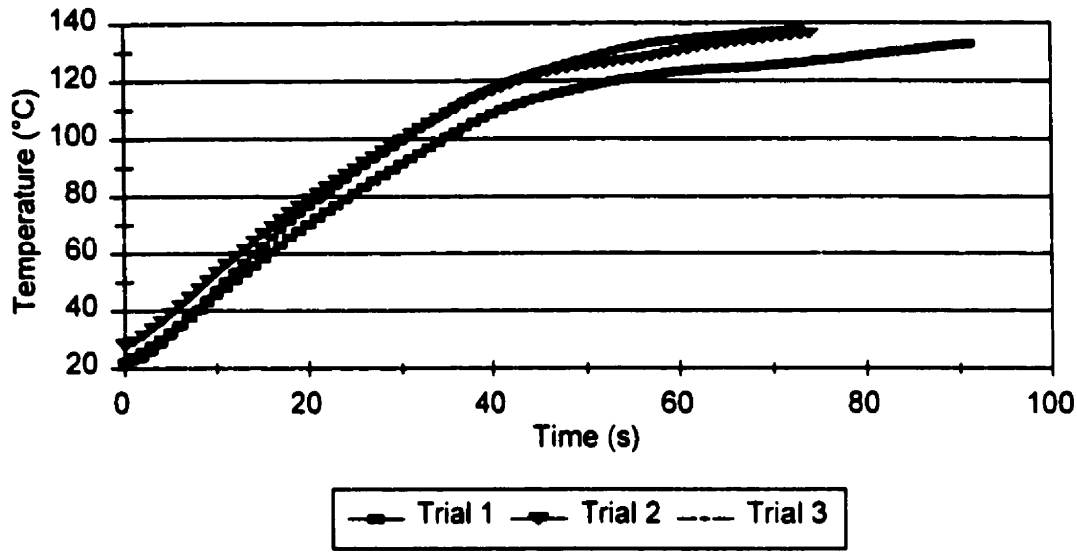


Figure 11 Average temperature history for each trial for samples at 26% mc wb and micronized at 16.8 kW/m^2.

All trials for each of the six treatments show similar heating patterns. The 26% mc treatment at 23.2 kW/m^2 had a difference of approximately 20°C between trials 1 and 3, possibly due to the warming up of the lamp in trial 1; it took about 25 s longer than trial 3 to reach 145.6°C. Graphs c-f in Appendix E show good agreement within different trials. At 12.3 kW/m^2 there is a smooth heating curve slightly changing slope at 10 and 50 s, whereas 16.8 kW/m^2 has a noticeable change in slope between 45-50 s, after which heating occurs at a slower rate. The 23.2 kW/m^2 intensity has a change in slope just before the 30 s mark, except for trial 3 at 28% mc, where it occurs at about 25 s.

Table 4 summarizes the maximum temperatures reached within the kernel, and the length of time the micronization process took to turn the kernels brown.

Table 4 Kernel Characteristics During Micronization

Intensity (kW/m^2)	Moisture Content (% wb)	Time of Browning (s)	Total Time of Micronization (s)	Maximum Temperature (°C)
12.3	28	61±1	104±16	138.3±7.4
	26	58±2	88±13	137.0±2.4
16.8	28	48±4	93±9	147.9±3.5
	26	48±3	79±8	135.8±2.2
23.2	28	36±1	42±6	129.5±4.7
	26	38±2	58±12	142.0±3.7

The time needed to turn the kernels brown increased with decreased intensity, as expected, though no concrete conclusions can be made with regard to the initial moisture content. Each time the lamp was lowered by 2 cm, the time for browning to occur decreased by approximately 12 s, or 19-23%. The total time required to fully micronize the samples also increased with decreased intensity. Comparing the 26% mc treatments, at 16.8 kW/m^2 it took 20 s longer for complete micronization of the samples than the samples micronized with 23.2 kW/m^2. It required 10 s more for the samples mirconized at 12.3 kW/m^2 compared with 16.8 kW/m^2. The moisture content also affected the total time of micronization. The greater the initial moisture content, the longer the time required for micronization. This could be due to the water heating up first then vaporizing. The exception, however, is the 16.8 kW/m^2 trials. Because the maximum temperature for the 28% mc trial is comparatively low, the full micronization time might not have been accomplished, however, by inspection they had appeared to be processed.

The temperature change per second was also determined (Figure 12 and Appendix F), as this allowed for an easier comparison between each treatment, as well as each trial. For example, the temperature history (Appendix Eb) for the 28% mc treatment at 23.2 kW/m^2 indicates that trial 1 had a slower heating time. However, it has a heating pattern similar to the other two trials when the rates of temperature change per second were compared.

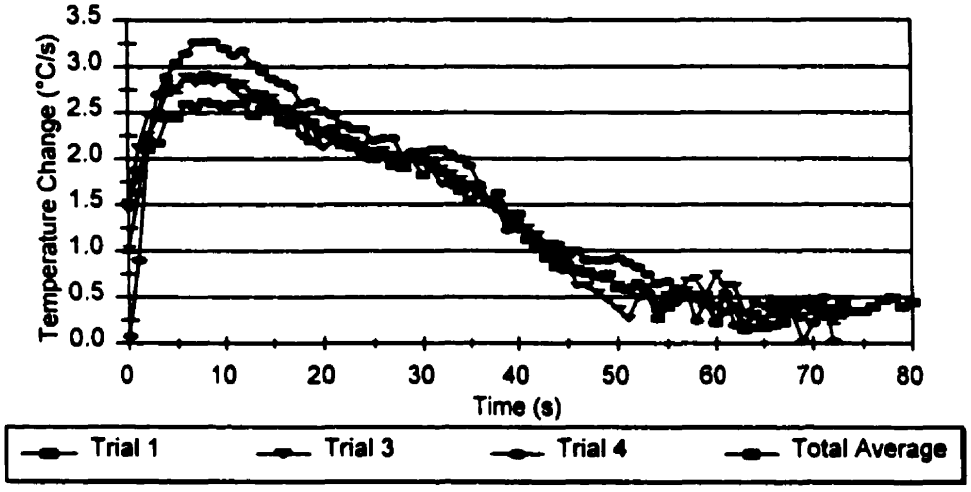


Figure 12 Temperature change per second during micronization for sample treated to 26% mc wb at 16.8 kW/m^2.

With the exception of the 26% mc treatments at 23.2 and 16.8 kW/m^2, the trials for each corresponding treatment are within a 0.5°C. Again, trial 1 of the 23.2 kW/m^2, 26% mc treatment is low, possibly due to an unusually cold room temperature, resulting in a lower initial lamp temperature, or just because it may have been the first trial run. Table 5 outlines the results of time of the greatest temperature change, the maximum temperature change rate, and the slope of the heating curve after the peak.

Table 5 Temperature Changing Characteristics

Intensity (kW/m^2)	Moisture Content (%)	Time of the Greatest Temperature Change (s)	Maximum Temperature Change per Second (s)	Slope of Curve after the Peak
12.3	28	9	2.3±0.1	- 0.03
	26	9	2.5±0.0	- 0.04
16.8	28	7	3.1±0.1	- 0.07
	26	8	2.9±0.3	- 0.06
23.2	28	6	4.3±0.1	- 0.10
	26	5	3.8±0.8	- 0.14

The greater the heat intensity at the pea surface, the greater the maximum temperature change which occurred at an earlier time. In addition, the slope of the pea heating curve after the peak decreases with decreased intensity confirming heating takes longer.

5.1.5 Mass Analysis - The moisture ratios (MR) of each treatment were determined (Figure 13) using

$$MR = \frac{M(\tau) - M_e}{M_o - M_e}$$

where:
MR = Moisture Ratio
$M(\tau)$ = moisture content at time, τ
M_e = equilibrium moisture content, 7.5%*
M_o = initial moisture content

* This value was determined at a heat intensity of 16.8 kW/m^2

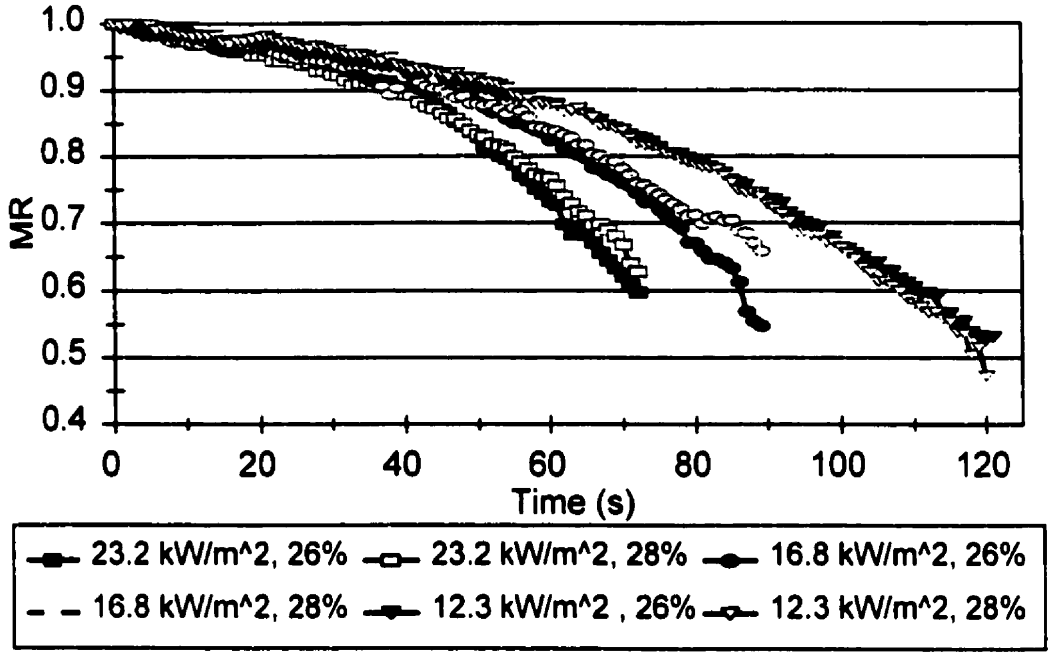


Figure 13 Moisture ratio histories of all six treatments during micronization

For the first 20 s of micronization, all treatments follow a similar pattern, after which the moisture ratio for the higher intensities drop quicker than the others. This coincides with the fact the higher intensities heat the product faster and therefore, evaporation of the water within the kernels would evaporate at a faster rate. At 60 s, the MR averaged for both moisture contents is 0.75±0.02, 0.83±0.01, and 0.88±0.00 for 23.2, 16.8, and 12.3 kW/m^2, respectively. This indicates a significantly small effect from the different initial moisture contents. The different treatments reached a MR of 0.7 at 62 and 66 s, 77 and 81 s, and 95 and 93 s for 26% and 28% moisture contents at 23.2, 16.8, and 12.3 kW/m^2, respectively.

Equations were fit to the MR curves for all treatments in the form of:

$$y = y_o + ax + bx^2 + cx^3$$

where:

y = moisture ratio
y_o, a, b, c = variables
x = treatment time

Table 6 lists the coefficients for each treatment, and Appendix G gives the graphical fit of the curves.

Table 6 Variables for MR Histories for Various IR Treatments

	26% mc			28% mc		
Variable	23.2 kW/m^2	16.8 kW/m^2	12.3 kW/m^2	23.2 kW/m^2	16.8 kW/m^2	12.3 kW/m^2
y_o	1.030	1.003	0.990	1.004	0.991	0.996
a ($x10^{-3}$)	-6.953	-2.781	-0.798	-3.021	0.939	0.605
b ($x10^{-5}$)	23.72	4.421	-1.200	4.435	1.894	1.560
c ($x10^{-6}$)	-3.407	-0.770	-0.114	1.022	-0.141	-0.117

Also, moisture content histories (Figure 14) show that the final moisture contents are within approximately 3% for the various treatments. This is interesting to note because this was not an initial determining factor in the length of micronization, but rather was the final temperature.

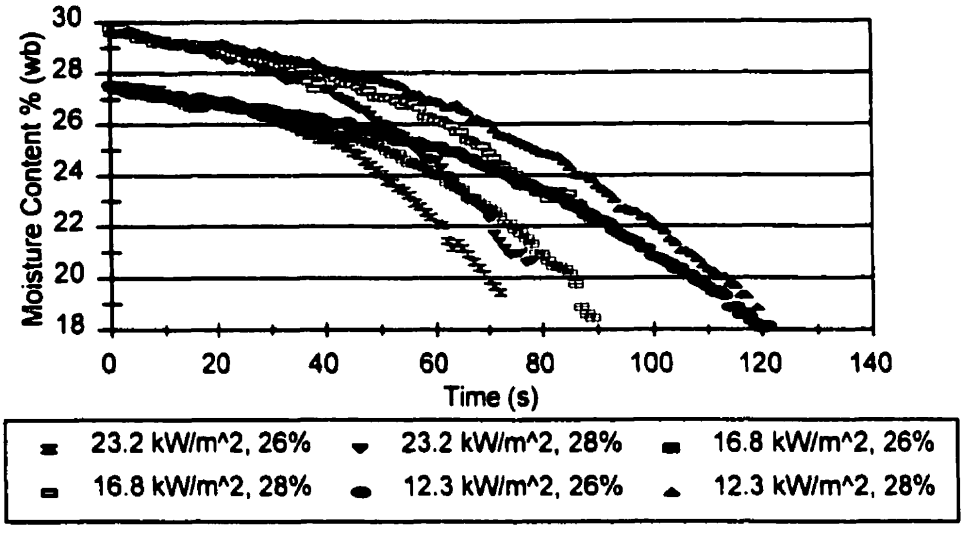


Figure 14 Moisture content history of all six treatments during micronization

5.2 Digestibility Determination

5.2.1 Dry Matter Digestibility - The overall dry matter digestibility was measured using *in vitro* digestibility tests and the amount of gelatinized starch was measured with the DSC7. As well, the nutritional components of the material, mainly protein and lipids, were determined to ensure the micronization process did not have a negative effect.

The dry matter digestibilities for the 26% mc treated samples and a reference sample are given in Table 7.

Table 7 Dry Matter Digestibility

Treatment (Intentsity kW/m²; %mc)	Dry Matter Digestibility (%)
Reference	45.3†
16.8; 26	71.4±0.8
23.2; 26	72.7±1.5

† one sample tube broke during digestion leaving only one sample

The different heat treatments were not statistically significant for increasing dry matter digestibility. The 23.2 kW/m^2, 26% mc treatment bag had a tear, leaking out some of the dry matter after freeze drying occurred. This accounts for the 1.5% standard deviation in this treatment as the dry matter mass was lower than expected. Overall, the heat treatment increased digestibility of the dehulled yellow peas by an average of 63.0%.

5.2.2 Digestible Proteins - The amount of protein digested *in vitro* was determined using the Kjedahl Method (N X 6.25). Table 8 shows the digested protein as the percentage of total protein in the peas, and the calculations for digestible protein determination are provided in Appendix H.

Table 8 Digestible Protein Determined Using Kjeldahl

Treatment (Intensity kW/m^2; %mc)	Digestible Protein (%)
Reference	82.4[†]
16.8; 26%	88.3±1.5
23.2; 26%	87.7±0.8

[†] one sample tube broke during digestion leaving only one sample

Again, the different heat treatments are not statistically different for the amount of protein available for digestion, though there is an overall improvement for the treated samples compared with the non-treated sample of approximately 7%.

5.2.3 Differential Scanning Calorimeter - When a sample is run in the DSC7, a graph indicating the melting points of the various components i.e. starch, protein, and lipids is produced. This graph indicates the melting-point temperature and the

amount of energy required (J/g) to melt each component, thus providing a comparison of the remaining nutrients in the non-micronized and micronized samples. Figure 15 represents a typical characteristic found in the literature (Nielsen 1994), and Figure 16 represents a typical characteristic from the pea samples run in the DSC7 in Biosystems Engineering. For confirmation purposes, a sample was run on a Differential Scanning Calorimeter (General V2.2A DuPont 9900) in the Department of Food Sciences. These results, illustrated in Figure 17, were similar to those obtained on the DSC7. The DuPont 9900, however, was not as sensitive as the DSC7, and therefore, produced smoother curves. Selected graphs from the DSC7 can be found in Appendix I.

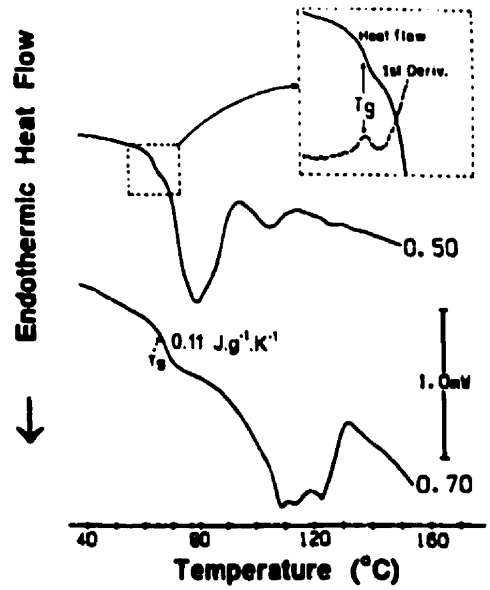


Figure 15 DSC thermal curves of a granular starch (at two different weight fractions) showing a glass transition (T_g) and melting transitions (Nielson 1994)

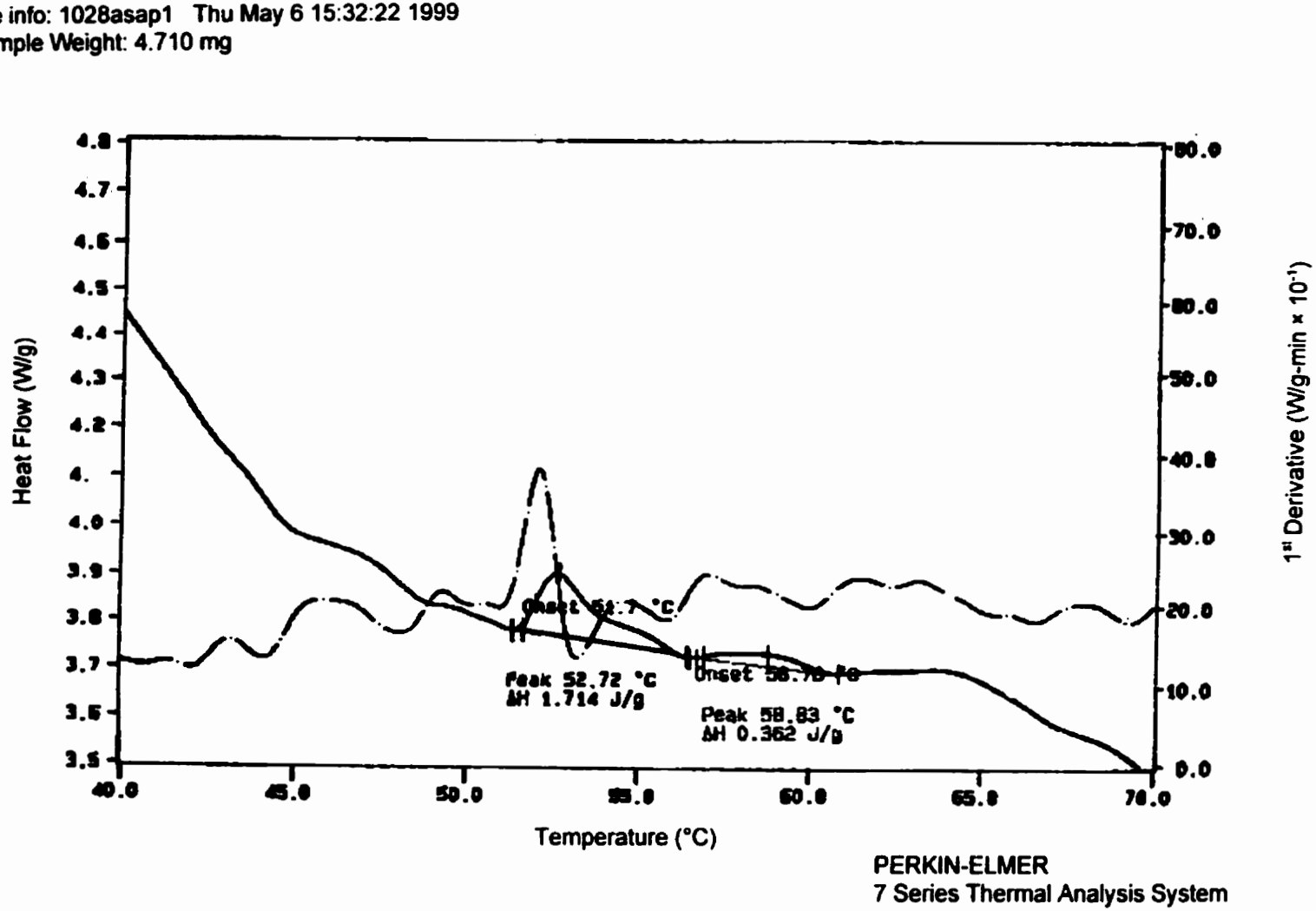


Figure 16 Typical melting profile for processed pea flour using the DSC7. The solid line represents the heat flow (W/g), and the broken line represents the 1st Derivative (W/g·min×10^{-1}).

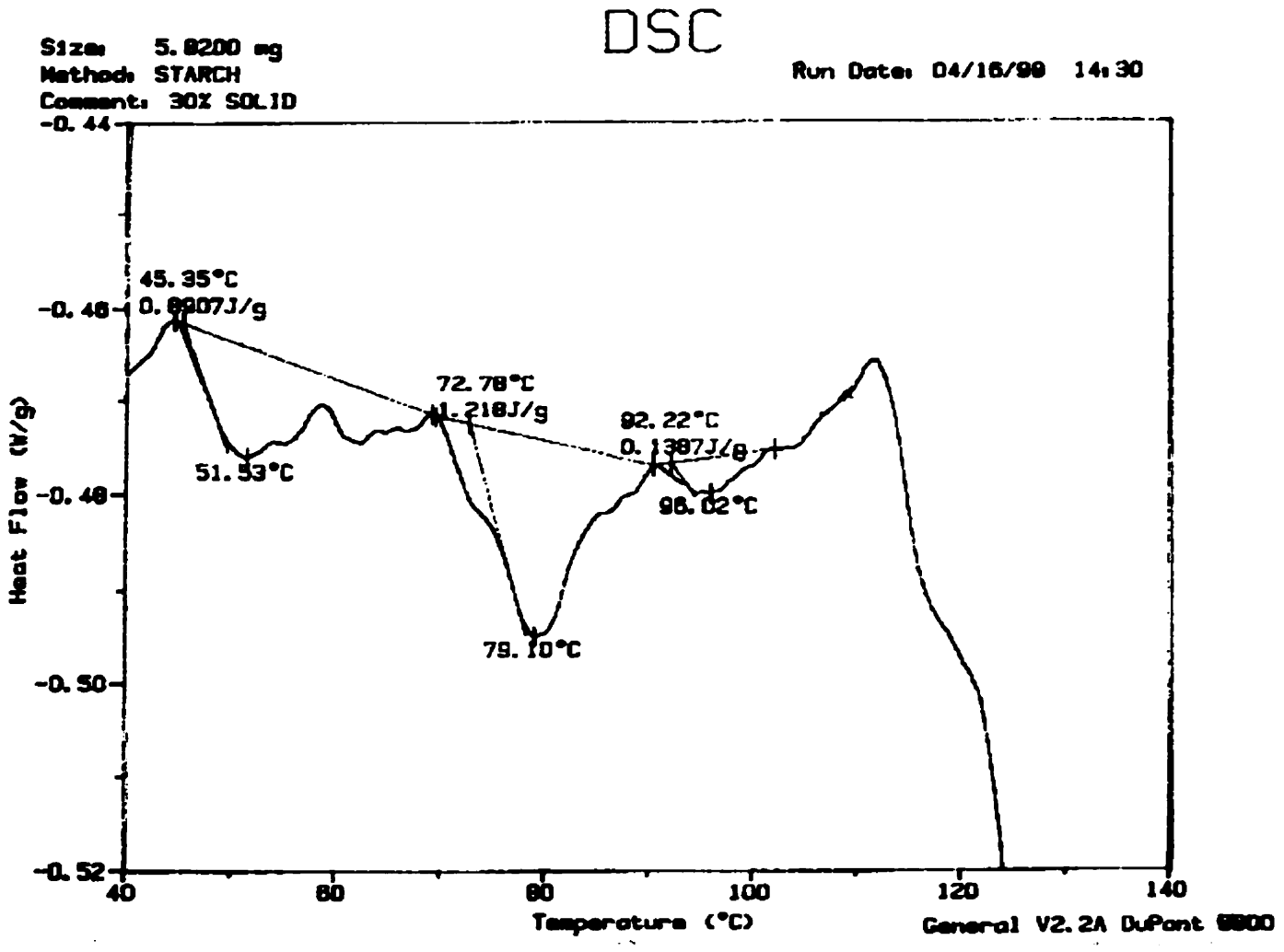


Figure 17 Typical melting profiles for processed pea flour using DuPont 9900

The temperature at which the melting takes place distinguishes between the components, but moisture brings down the gelatinization temperature of starch – instead of a 200°C gelatinization temperature with little or no available water, the melting temperature of the starch is reduced to 65°C, when water was added to make a 30% solid mix. Solid fractions below 50% show little variability in the onset temperatures. Increased availability of water also promotes metastability which yields a single melting point (Walter 1998). Solid mixes above 50% could be misleading, as the shoulder becomes the main peak. Figure 18 illustrates the effect of water on the DSC7 profiles. However, the literature indicates that researchers use different water to solid ratios (Cenkowski and Sosulski 1997). Trials were performed with pure waxy maize, resulting in an onset melting temperature of 65.4°C and requiring 13.75 J/g (Appendix J). Using this information, the starch in the pea flour samples could be distinguished.

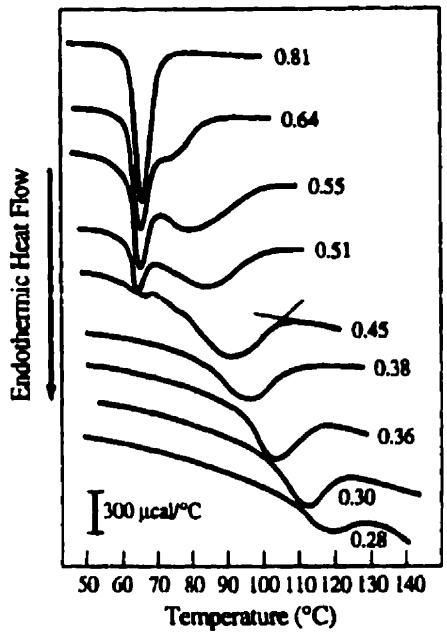


Figure 18 The effect of different water fractions on starch gelatinization of potatoes (Nielson 1994).

Starch - Table 9 displays the amount of energy required to gelatinize the remaining starch in the processed and unprocessed kernels. The change in enthalpy, ΔH (J/g), represents the sum of all the peaks in the temperature range for which gelatinization of starch occurs (approximately 49°C to 55°C), since metastability was not apparent. The onset temperature was used rather than the peak temperature because of the varying interpretation of this temperature. Some researchers interpret it as the point when all the material is melted, but others disagree and believe it is unreliable because it is greatly influenced by the scan rate and sample size (Nielsen 1994). Since the sample sizes varied by ±2.0 mg, the onset temperatures are displayed.

Table 9 Amount of Energy Required to Gelatinize Starch

Treatment (Intensity kW/m^2; %mc)	ΔH (J/g)	Onset Temperature (°C)
23.2; 28	0.07±0.06	54.6±0.2
23.2; 26	0.11±0.06	53.4±6.3
23.2; 24	0.11±0.03	52.6±2.0
16.8; 28	0.19±0.14	51.3±4.6
16.8; 26	0.32±0.20	51.0±3.5
12.3; 28	0.25±0.16	51.1±5.8
12.3; 26	0.37±0.28	49.2±3.6
reference pea	5.08±0.45	63.4±2.4
waxy maize	13.75	65.4

The reference pea flour had an onset temperature of 63.4°C, within 3.2% of the pure waxy maize. As a result of micronization, the onset temperatures of the samples were lower than the reference samples. This is due to the reorganization of the starch during the heat treatment, making it available for gelatinization at a lower temperature. Reducing the heat intensity resulted in a lower onset temperature. This is the reverse of what was expected since the reference sample had the highest onset temperatures. This could be a result of high standard deviations but could also be an effect of heat treatments – much like that shown by Slominiski (1997) with digestible protein as a function of heat treatment temperature. If an even lower intensity had been tried, the onset temperature might have started to increase. Another explanation could be due to varying moisture contents, though all samples were left to equilibrate in the same conditions.

In general, these results show the higher the intensity of the infrared heat source, in conjunction with a high moisture content the less energy required to melt the starch, indicating more starch was gelatinized. The moisture content seemed to have a greater effect with the lower intensities, though the standard deviations also increased. This increase in ΔH is explained because micronization is dependent on the available moisture. The more moisture available, the greater the vapor pressure and inter-molecular friction, thus aiding the breaking of the starch's elastic wall. The reference pea flour sample required significantly greater energy, 5.08 J/g, to gelatinize the available starch, thus demonstrating the effect of micronization.

The effect of retrogradation, or the reforming of starch after time, is illustrated in Table 10. The sample that was tested in the DSC7 immediately after micronization needed 0.25 J/g to melt the starch and 0.31 J/g and 0.32 J/g was required for the samples tested 24 h and 14 days after micronization, respectively, indicating the reforming of starch is most prominent within the first 24 h.

Table 10 Effect of Retrogradation of Starch

Treatment (Intensity kW/m²; %mc; time after micronization)	ΔH (J/g)	Onset Temperature (°C)
16.8; 26; asap†	0.25	54.3+
16.8; 26; 24 h	0.31	58.2+
16.8; 26; 2 wk	0.32	50.5

† as soon as possible
+ indicates only one peak on graph (metastability)

Lipids and α-amylase - The results listed in Table 11 show an increased amount of energy required to melt the lipids in the treated samples compared with the reference sample. This is due to the mobilization of lipids that occurs during micronization, making them more readily available during the melting process. The effect of retrogradation is not relevant for lipids in terms of the amount present, because once the material is cooled, the lipids reform immediately, however, once reformed they melted at a different temperature due to this restructuring. For example, the reference sample had a melting onset point of 94.9°C for lipids, whereas the 23.2 kW/m^2, 28% mc sample had a melting point of 95.7°C. On the other hand, the 12.3 kW/m^2 , 28% mc sample started to melt at 91.6°C.

Table 11 Amount of Energy Required to Melt Lipids

Treatment (Intensity kW/m^2; %mc)	ΔH (J/g)	Onset Temperature (°C)
23.2; 28	1.81	95.7
23.2; 26	0.84±0.12	91.3
16.8; 28 [a]	–	–
16.8; 26	1.08±1.06	95.1±3.8
12.3; 28	1.46±0.06	97.5±3.8
12.3; 26	1.15±0.67	91.3±4.5
reference pea	0.39±0.13	94.9±1.7

[a] no gelatinization was found in this range except a ΔH of 7.8 at 96.2°C, which was disregarded as experimental error after the Q-value test was performed (Neilsen 1994).

5.3 Observation

5.3.1 Storage of Micronized Dehulled Yellow Peas - As an observation, the storage of the micronized kernels was monitored. Though no structural tests were performed, kernels that had been stored up to two years, in the dark and at room temperature, had no difference in appearance than those that had just been micronized. In almost all of the tests that were performed, the kernels were cooled down before placing into a plastic bag, which also allowed for any moisture on the surface of the peas to evaporate. The peas that were tested for mass measurements, however, were not allowed to cool down as moisture would have escaped. After about 4 wk green mold started to appear on these samples, indicating the amount of water remaining on the surface of micronized material would support bacterial fungi.

6. Moving Multi-Lamp Micronizer

6.1 Design Specifications

With the information from the previous results, specifications were set out for the design and fabrication of a movable multi-lamp micronizer (MLM). Since this machine was designed to be used in a household or small company, it was also important to take the machine's physical factors into account, for example ease of storage and portability.

6.1.1 Heat Intensity - The optimum heat intensity was determined based on several factors: the uniformity of the lamp; the temperature and moisture changes within the kernels; and how the IR treatment affected digestibility.

Digestibility - Firstly, IR treatment is known to increase dry matter digestibility, but the amount of starch that was gelatinized needed to be determined. It was also necessary to make sure the digestible protein was not adversely affected by the process, since the type of heat treatment could adversely affect digestibility if the treatment was too harsh. Thus, the optimum heat intensity was determined such that the product not only increased the digestibility of the dry matter and the starch but the amount of digestible protein increased as well. Both the 16.8 and 23.2 kW/m^2 heat intensities increased digestible protein in addition to increasing the dry matter and starch digestibility, but the 23.2 kW/m^2 heat intensity only required half of the amount of energy to gelatinize the starch when compared with the 16.8 kW/m^2 heat intensity. This indicates there was less starch available.

Heat Uniformity - As previously discussed, the middle area (positions 3-6, a 9 cm length, Figure 7) under the IR unit receives a greater and more uniform heat intensity than the ends, staying within 2.3°C. The 12.3 kW/m^2 heat intensity has the greatest uniformity with a variance of only 0.5°C in this area. For these reasons, this middle area under the lamp is recommended for processing uniformity, but to process a larger volume of peas, the whole length of the lamp can be utilized if a vibrating tray was implemented.

Temperature and Moisture Changes - The greater the heat intensity, the quicker the kernels attained the final heating temperature and the faster they lost moisture, therefore, the 23.2 kW/m^2 heat intensity is optimum in terms of heating rate, while at the same time not burning the product.

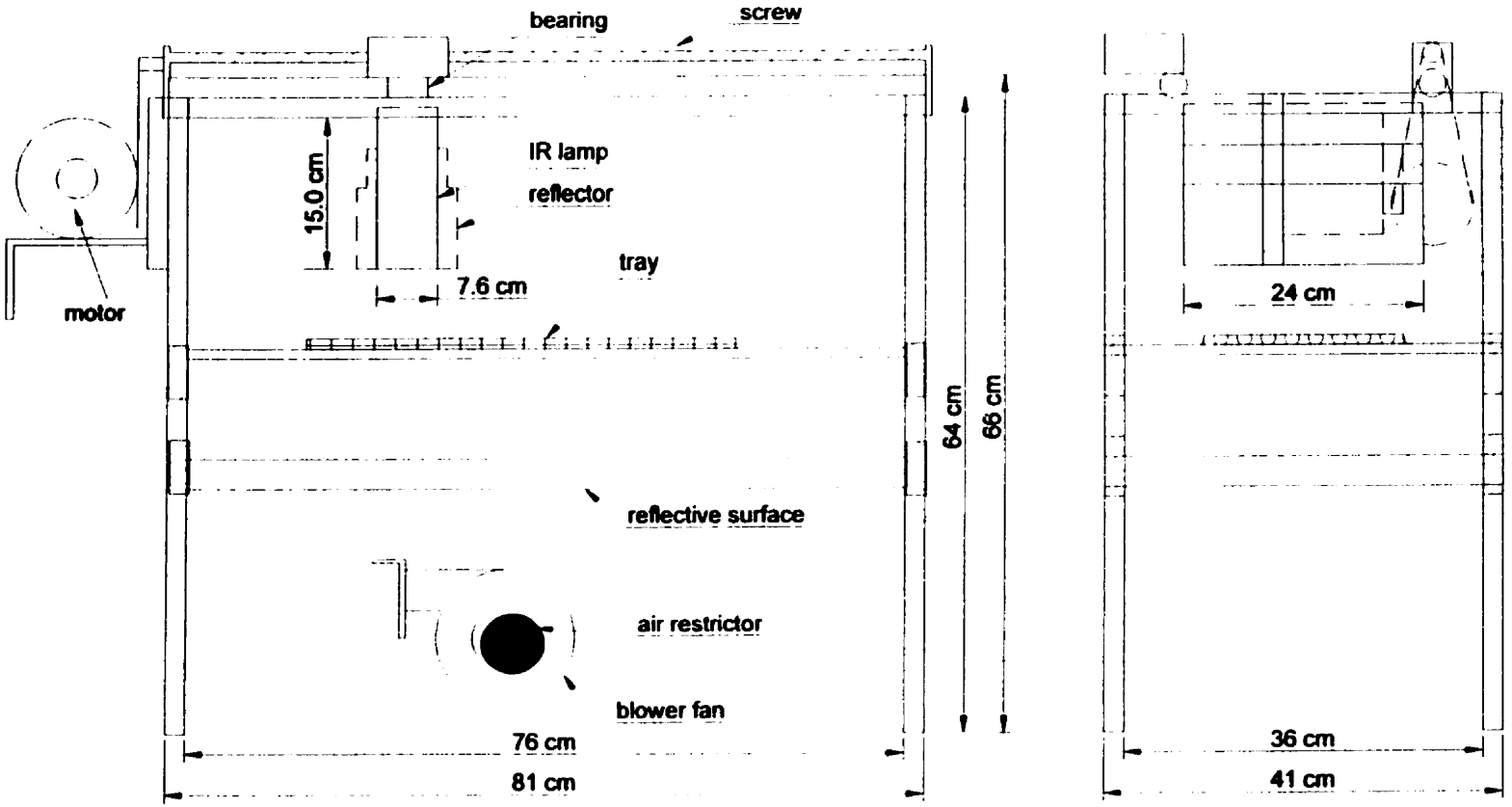
Velocity - Ideally, the kernels would move on a vibrating conveyor belt under a stationary lamp. For simplicity in fabrication, however, the micronizer was designed such that the IR unit would move along a stationary vibrating tray. The velocity of the moving IR unit should compare with the length of optimum heating for the stationary unit. Assuming the heat from the lamp emits to a 120 mm width (using the light emitted from the lamp as a guide to where the heat hits the material), and knowing it takes approximately 80 s to process the material at a height of 10 cm and 60 s at 8 cm, the unit should travel at a velocity of 1.5 mm/s (9.0 cm/min) or 2.0 mm/s (12.0 cm/min).

Other Factors - The IR micronizing unit should have a reflective surface beneath the material tray. The reflective surface prevents the table top from burning, and any heat reflected from the surface acts as a convective heater for the material. As previously mentioned, the material tray should vibrate to ensure uniform heating of the kernels. A blower fan Model KB 4553 (Research Inc., Minneapolis, MN) was installed to provide forced cooling to the Pyropanel. This allows for convective heat flow and aids in the lifetime of the of the IR bulbs.

Taking these factors into consideration, the 23.2 kW/m^2 heat intensity is optimum. The temperature is within 2°C under the middle area of the lamp, and within 10°C overall. The final processing temperature for the 26% mc samples was 6°C higher than the 16.8 kW/m^2 treatment, but reached it 20 s faster than the 16.8 kW/m^2 treatment without sacrificing any nutritional components. In fact, not only was the dry matter digestibility increased, but more starch was gelatinized than at any other intensity and the digestible protein increased. However, the moveable machine was designed to accommodate the 23.2 and 16.8 kW/m^2 heat intensities as the 16.8 kW/m^2 heat intensity had almost the same effect on digestibility as the greater heat intensity, but required more energy to gelatinize the remaining starch. Figure 19 shows the specifications for the moveable IR micronizer.

The frame is made from 2.54 cm-dia steel tubing and the IR lamp is powered by a variable speed, reversible, fractional horsepower gearmotor (Bodine Electric Company, Chicago, Il), 1/20 hp and 43 rpm. The desired range of speed is obtained using a 30 and 10 tooth gear in combination. The lamp is connected to bearings that travel along a 5.1 threads/cm (13 threads/inch) screw that guide the lamp on

two 2.54 cm-dia steel rods. The reflective surface and tray heights are adjustable, and the tray, which is perforated metal, is removable to accommodate a change in size. A 7.0 cm-dia corrugated flexible duct connects the blower fan to the air plenum on the lamp.



a) Front View

b) Side View

Figure 19 Sketch of the MLM and the blower fan connected to the IR lamp. A 7.0 cm-dia corrugated flexible duct connects the blower fan to the air plenum on the lamp.

6.2 Multi-Lamp Micronizer Results

The MLM was operated at the previously recommended intensity and speed of 16.8 kW/m^2 and 9.0 cm/min, respectively, and the gap for airflow into the fan was opened to result in an air speed of 1.7 m/s. The temperature histories of the kernels across the length of the IR lamps were recorded (Figure 20). The overall temperature distribution varied by 10°C, an improvement from the previous results with the stationary micronizer. The maximum temperature reached only 79.4°C, 60°C lower than the temperature attainable with the stationary lamp. This temperature difference could be attributed to the forced convection effect from the blower fan. The fan is recommended for extended use of the IR lamps, thus, the heating parameters had to be changed to accommodate this cooling effect (Research Inc. 1994).

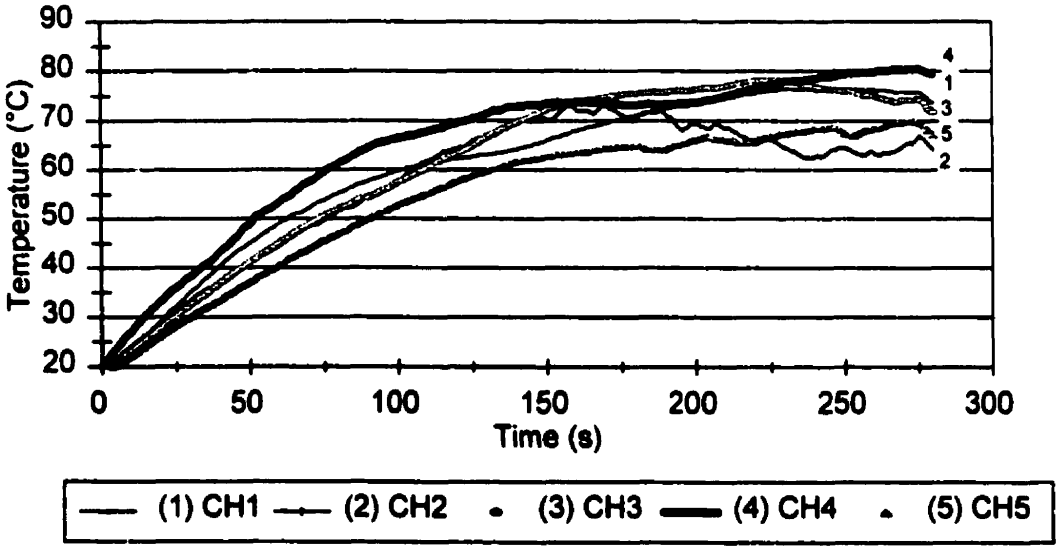


Figure 20 Temperature history of kernels using the MLM at 16.8 kW/m^2, 9.0 cm/min speed, and an air speed of 1.7 m/s. Channels 1-5 were located throughout the length of the tray.

The air gap for the fan was closed to 1 cm (airspeed of 1.3 m/s), the smallest manufacturer-recommended gap (Research Inc. 1994), the operating speed of the MLM was slowed to 6.2 cm/min, and the temperature histories were again recorded (Figure 21). The average maximum temperature reached was 95.8±2.7°C, which resulted in a good correlation of temperatures across the lamp but a low maximum temperature. The drop in temperature for channel 4 could be attributed to the moisture migration taking place withing kernel. As the moisture is passing over the thermocouple, it would have a cooling effect.

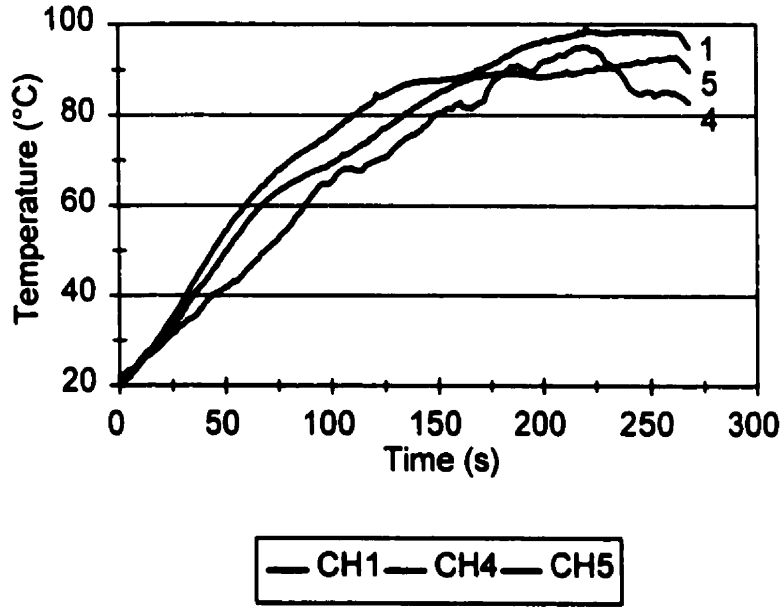


Figure 21 Temperature history of kernels using the MLM at a height of 16.8 kW/m^2, lamp speed of 6.2 cm/min, and an airspeed of 1.3 m/s.

Because the heat intensity was 16.8 kW/m^2, and the maximum temperatures were still not above 100°C, the intensity was increased to 23.0 kW/m^2 (equivalent to the 8 cm height for the stationary lamp). Previously, it was stated that both

intensities were recommended, but the 23.0 kW/m^2 intensity might be too harsh for the surroundings – based on the stationary lamp results. However, since the kernels were not sitting directly on a table top, but were suspended thus leaving an air gap between the tray and the reflective surface, the 23.0 kW/m^2 intensity was appropriate and would not burn the reflective surface. The intensity hitting the kernels was confirmed using the same method described for the stationary lamp. Figure 22 shows the temperature history for this trial (T1) which had a steady heating characteristic, and the maximum temperature for the kernels across the lamp was 100.7±2.3°C, only a 4.9°C increase from those results in Figure 21. The lamp speed was then slowed to 3.9 cm/min (Figure 22, trial 2 (T2)). The maximum temperature was 103.1±2.1°C; a small increase for decreasing the time of processing. The maximum temperature of kernels was usually reached when the kernels were directly below the middle of the lamp. In these trials, the maximum temperature was maintained as the lamp passed over the kernels. The first area heated consistently reached its maximum temperature *after* the lamp had completely passed over it. The fact that the temperature within the kernels was still increasing after the lamp passed over the kernels and that the maximum temperature was maintained indicate that gelatinization could still be taking place within the peas. Thus, as long as the kernels are maintained at a high temperature, the 140°C maximum temperature might need not be reached. This reflects the results found by Kouzeh-Kanani et al. (1981). Nonetheless, trials were continued to reach a higher temperature, but none were successful.

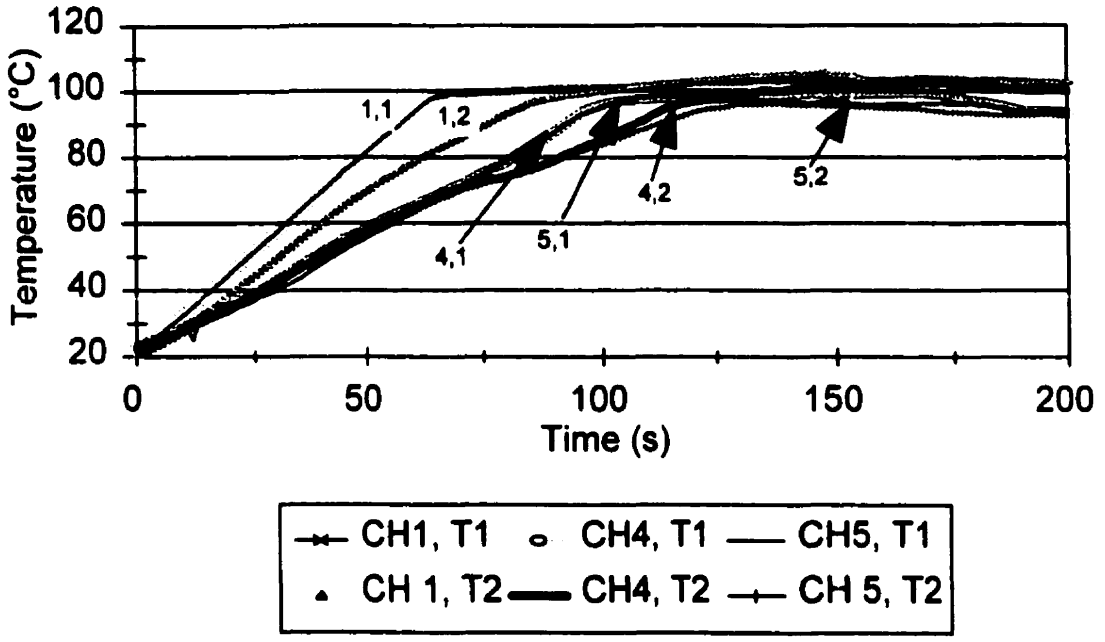


Figure 22 Temperature histories of kernels using the MLM at 23.0 kW/m^2 and an airspeed of 1.3 m/s. T1 indicates a lamp velocity of 6.2 cm/min and T2 indicates a lamp velocity of 3.9 cm/min.

A trial was performed without the fan and at a lamp speed of 6.2 cm/min to determine the fan's effect. The kernels only reached a temperature of 114.0°C and they were charred.

Finally, trials were repeated for the following conditions: 23.0 kW/m^2; IR lamp speed of 6.2 cm/min; and an airspeed of 1.3 m/s. Figure 23 is a representation of the temperature history. An average maximum temperature of 105.4±3.4°C was attained by the kernels. The first thermocouple location had the lowest maximum temperature, but the temperature was consistently maintained within 5°C for about 45 s. The kernel that was positioned last under the lamp maintained its maximum temperature for about 30 s. The temperature remained above 95°C for 45, 80, and 130 s for the first, second, and third thermocouple locations, respectively. So, even

though the maximum temperature was not maintained within 5°C for the latter positions, it remained above 95°C for an extended period of time.

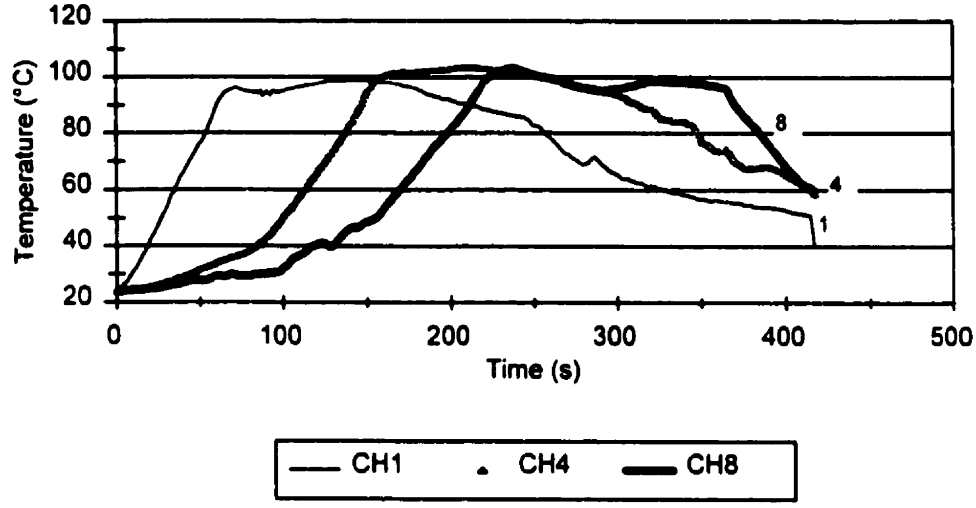


Figure 23 Average temperature histories of kernels processed with the MLM at three locations.

6.2.1 Digestibility Determination for the Multi-Lamp Micronizer - The kernels did not reach an expected temperature of 140°C so the starch gelatinization was confirmed with DSC7 tests. These trials were similar to the previous tests, except the scanning rate was lowered to 5°C/min to obtain a smoother curve. Since the scanning rate affects the ΔH value, the reference sample had to be repeated for comparison. Table 12 lists the melting points and ΔH values of starch from the DSC7 profiles.

Table 12 Onset Temperatures and ΔH values of Starch Processed with the MLM

Sample	Onset Temp (°C)	ΔH (J/g)
MLM treatment	63.5±0.5	1.95±0.22
Reference pea	54.7±4.7	3.72±0.25

The reference pea required 3.72 J/g of energy to gelatinize the starch in the pea flour. The previous results had a ΔH of 5.08 J/g, 27% higher than this trial, perhaps indicating the difference in scanning rate. It was noted that when the samples were prepared the water did not disperse throughout the pea flour as easily as it had in the past. Instead, the tip of a micro-syringe was used to stir the water into the pea flour.

The MLM resulted in a decrease in the amount of energy required for gelatinization. The reference pea flour required 3.72 J/g and the treated pea flour needed 1.95 J/g, a 47.5% decrease, despite having a lower maximum temperature attained during the micronization process than the stationary lamp.

7. CONCLUSIONS

Micronization is an effective heat treatment for increasing digestibility and decreasing cooking time of dehulled yellow peas. Because of these benefits, a household IR micronizer, run on electricity, was designed and fabricated.

This study resulted in the following conclusions:

1. The theoretical amount of heat energy emitted from the lamp that reached the material decreased exponentially with increased height between the heat source and the material, and this was confirmed. The overall uniformity of heat energy reaching the material, however, was not affected. Of the energy emitted from the IR lamp, 38, 29, and 22% reached the pea surface at lamp heights of 8, 10, and 12 cm, respectively.

2. The greater the IR lamp intensity the quicker the micronization process occurred. The 23.2 kW/m^2 heat intensity took only 60 s to micronize and had no adverse effects on digestibility, and the 16.8 kW/m^2 intensity took 80 s for micronziation and had comparable digestibility results. For a stationary lamp, the lower intensity is recommended for safety purposes.

   The initial moisture content did not have any conclusive effect on the rate of heating of the material or the moisture ratio histories. Comparing the 26 and 28% mc the time of browning varied by a maximum of 3 s, and the total time of micronization varied by a maximum of 16 s. The maximum

temperature reached upon completion of micronization was greater with increased heat intensity but there was no absolute results in terms of moisture content – the maximum temperature for the 12.3 kW/m^2 heat intensity differed by only 1°C between the two moisture contents; at 16.8 kW/m^2 the 26% mc was processed to 12°C less than the 28% mc; and the 26% mc treatment was micronized 12°C more than the 28% mc treatment at 23.2 kW/m^2.

3. Micronization with the stationary lamp increased the dry matter digestibility 36% by: increasing the amount of available digestible protein by 7%; requiring at least 93% less heat energy to gelatinize starch in the peas; and re-organizing the lipid structure. In general, increased initial moisture contents resulted in increased gelatinization of the available starch. The effect of moisture content decreased with increased radiation intensity.

4. An electric-powered, portable multi-lamp IR micronizer was designed and fabricated to micronize peas at heat intensities of 16.8 and 23.2 kW/m^2, with the ability to adjust the heat intensity for future testing.

The average maximum temperature reached by the kernels was 105.4°C – approximately 35°C lower than the stationary lamp. As the lamp completely passed over the first heated area, the temperature within the kernels remained above 95°C (within 5°C of the maximum temperature) for

45 s, and the subsequent areas maintained a temperature above 95°C for 80 and 130 s.

With the moveable unit, the starch required 47.5% less heat energy than the reference sample to gelatinize, which was less than the stationary unit. This indicates a decrease in gelatinization when comparing the moveable lamp to the stationary lamp.

5. Approximately 4 wk after the micronization process, molding resulted when water was not evaporated from the surface of the kernels. Those kernels that were cooled after micronization and stored up to two years were not affected by mold growth and appeared to be in tact.

8. RECOMMENDATIONS

This study proved micronization was an effective means of heat treatment for dehulled yellow peas, and a feasible method of processing for small, rural-based, value-added companies. The following recommendations for further study were devised from this work:

1. Enclose the unit for improved safety precautions. The unit supporting the IR bulbs did not get hot enough to cause burning during these experiments. However, if the machine is run for hours at a time, it will get hot enough to cause injury if touched, therefore a guard or shield is recommended. Also, if the unit is enclosed greater efficiency of the system due to convection should be determined.

2. Eventually, a vibrating conveyor belt should be devised such that the material will be moving, and not the lamp. This would simulate the industrial-sized, gas-fired machines and would allow for easier cooling and packaging of the kernels.

3. Design a controlled unloading system to aid in the packaging process. After the peas have been micronized, it is important to cool them to prevent any packaging from melting as well as allowing the excess moisture to evaporate. The kernels being micronized first would have a chance to cool down, though last ones to receive the IR heat will still be hot and leaving the kernels on the

tray may stall processing. Therefore, a device should be designed to allow for sufficient cooling time as well as provide a simple emptying device for packaging the material.

4. Additional materials should be tested on this machine to determine their optimum processing characteristics while maintaining good digestibility.

5. The reason for the lower maximum temperature of the kernels should be determined, and further test the effects of high-temperature holding-times on gelatinization and efficiency.

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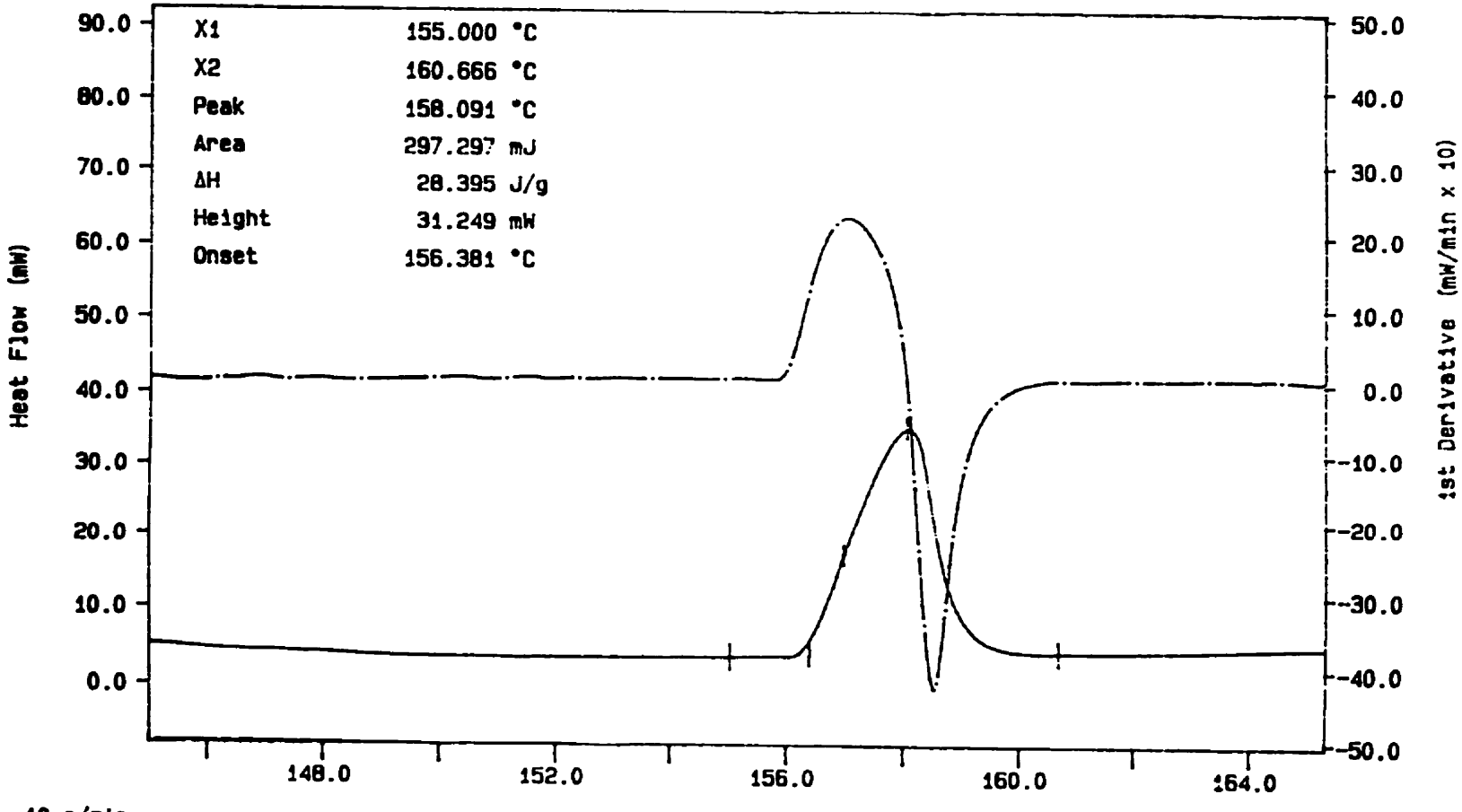

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APPENDIX B PREPARATION OF SOLUTIONS FOR DIALYSIS/DIGESTIBILITY EXPERIMENTS

0.05 M phosphate buffer pH 7.0

75.35 g of monobasic sodium phospate (monohydrate) was dissolved in 2.73 L distilled water (Solution A). 121.27 g of dibasic sodium phsphate was dissolved in 4.27 L distilled water (Solution B). The two solutions were mixed and 14.00 L distilled water was added to make 0.05 M buffer.

0.1 M phosphate buffer pH 7.0

5.38 g of monobasic sodium phosphate, monohydrate was dissolved in 250 mL distilled water (Solution A), and 8.66 g of dibasic sodium phosphate in 500 mL distilled water (Solution B). The two solutions were mixed and diluted to 1.0 L.

1.0 M HCl/54 mM NaCl solution

8.5 ml concentrated HCl (37%) was added to 3.2 g NaCl and made up to 1.0 L with distilled water.

Pancreatin solution

0.2 g pancreatin (P 7545, 8 x U.S.P., Sigma) was dissolved in 2.0 ml of 0.1 M phosphate buffer.

Materials:

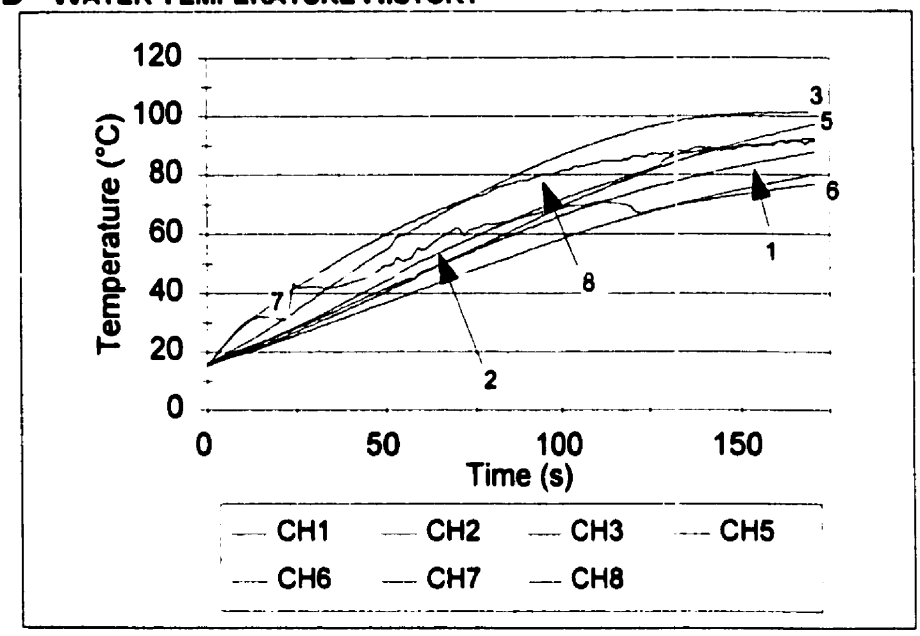
Spectrum Spectra/Por Molecularporous membrane tubing
Dialysis 45 mm tubing 100 ft flat
- Width: 45±2 mm
- Diameter: 29 mm
- Vol/Length: 6.4 ml/cm

APPENDIX C CONFIGURATION FACTOR FOR IDENTICAL, PARALLEL DIRECTLY OPPOSED RECTANGLES

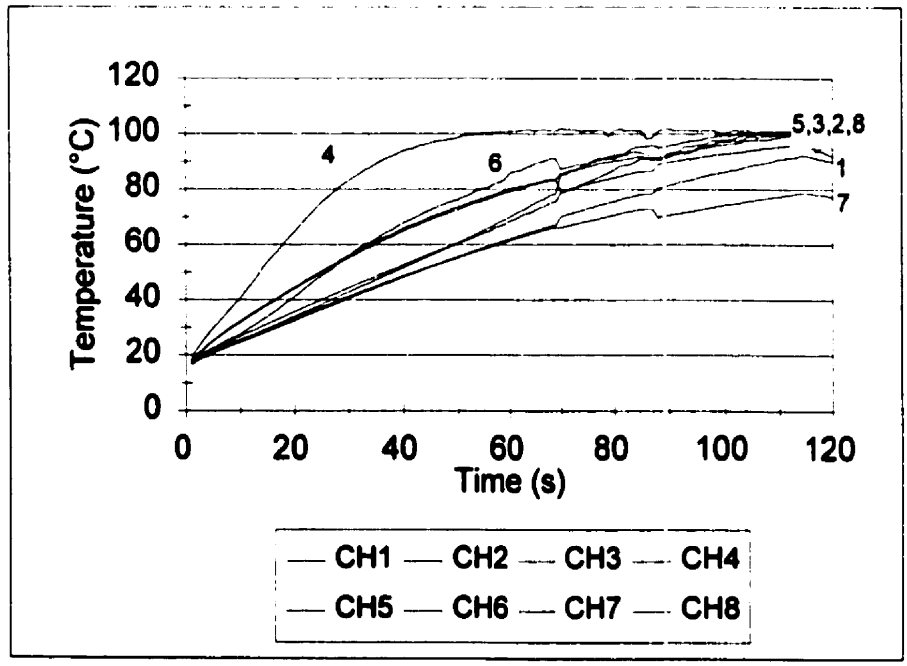
c[1] (cm)	X	Y	2/(pi*XY)	1st term	2nd term	3rd term	4th term	5th term	F(1-2)
0.25	30.40	72.00	0.0003	3.33	874.47	2564.66	46.75	112.10	0.96
0.50	15.20	36.00	0.0012	2.64	218.62	641.89	22.88	55.55	0.91
0.75	10.13	24.00	0.0026	2.24	97.17	285.81	14.92	36.70	0.87
1.00	7.60	18.00	0.0047	1.95	54.66	161.19	10.94	27.28	0.84
1.25	6.08	14.40	0.0073	1.74	34.99	103.50	8.56	21.62	0.80
1.50	5.07	12.00	0.0105	1.56	24.30	72.16	6.97	17.85	0.77
1.75	4.34	10.29	0.0143	1.41	17.85	53.26	5.84	15.16	0.73
2.00	3.80	9.00	0.0186	1.29	13.67	40.99	4.99	13.14	0.70
2.25	3.38	8.00	0.0236	1.18	10.80	32.58	4.33	11.57	0.68
2.50	3.04	7.20	0.0291	1.08	8.75	26.56	3.81	10.32	0.65
2.75	2.76	6.55	0.0352	1.00	7.24	22.10	3.38	9.29	0.62
3.00	2.53	6.00	0.0419	0.92	6.08	18.71	3.03	8.43	0.60
3.25	2.34	5.54	0.0492	0.85	5.18	16.06	2.73	7.71	0.57
3.50	2.17	5.14	0.0570	0.79	4.47	13.96	2.47	7.09	0.55
3.75	2.03	4.80	0.0654	0.74	3.89	12.27	2.25	6.55	0.53
4.00	1.90	4.50	0.0745	0.69	3.42	10.88	2.06	6.08	0.51
4.25	1.79	4.24	0.0841	0.64	3.03	9.72	1.90	5.67	0.49
4.50	1.69	4.00	0.0942	0.60	2.71	8.75	1.75	5.30	0.47
4.75	1.60	3.79	0.1050	0.56	2.43	7.93	1.62	4.97	0.45
5.00	1.52	3.60	0.1163	0.52	2.19	7.22	1.50	4.68	0.44
5.25	1.45	3.43	0.1283	0.49	1.99	6.62	1.40	4.41	0.42
5.50	1.38	3.27	0.1408	0.46	1.81	6.09	1.30	4.17	0.41
5.75	1.32	3.13	0.1539	0.43	1.66	5.62	1.22	3.95	0.39
6.00	**1.27**	**3.00**	**0.1675**	**0.40**	**1.53**	**5.22**	**1.14**	**3.75**	**0.38**
6.25	1.22	2.88	0.1818	0.38	1.41	4.85	1.07	3.56	0.36
6.50	1.17	2.77	0.1966	0.36	1.30	4.53	1.01	3.39	0.35
6.75	1.13	2.67	0.2120	0.34	1.21	4.24	0.95	3.23	0.34
7.00	1.09	2.57	0.2280	0.32	1.12	3.98	0.90	3.09	0.33
7.25	1.05	2.48	0.2446	0.30	1.05	3.75	0.85	2.95	0.32
7.50	1.01	2.40	0.2618	0.28	0.98	3.54	0.80	2.82	0.31
7.75	0.98	2.32	0.2795	0.27	0.92	3.34	0.76	2.70	0.30
8.00	**0.95**	**2.25**	**0.2978**	**0.25**	**0.86**	**3.17**	**0.72**	**2.59**	**0.29**
8.25	0.92	2.18	0.3167	0.24	0.81	3.01	0.69	2.49	0.28
8.50	0.89	2.12	0.3362	0.23	0.76	2.86	0.65	2.39	0.27
8.75	0.87	2.06	0.3563	0.21	0.72	2.72	0.62	2.30	0.26
9.00	0.84	2.00	0.3769	0.20	0.68	2.59	0.59	2.21	0.25
9.25	0.82	1.95	0.3982	0.19	0.65	2.48	0.57	2.13	0.25
9.50	0.80	1.89	0.4200	0.18	0.61	2.37	0.54	2.06	0.24
9.75	0.78	1.85	0.4424	0.17	0.58	2.27	0.52	1.98	0.23
10.00	**0.76**	**1.80**	**0.4654**	**0.16**	**0.55**	**2.17**	**0.49**	**1.91**	**0.22**

[1]The IR lamps are 2.0 cm above the bottom of the vertical shielding (Figure 6). All of the energy that is given off by the lamps passes through this opening (assuming reflectivity is 1.0), since it is the only opening, and it acts as the 'invisible source.' Therefore, the variable *c* is used as 4, 6, 8, and 10 cm for the respective lamp heights.

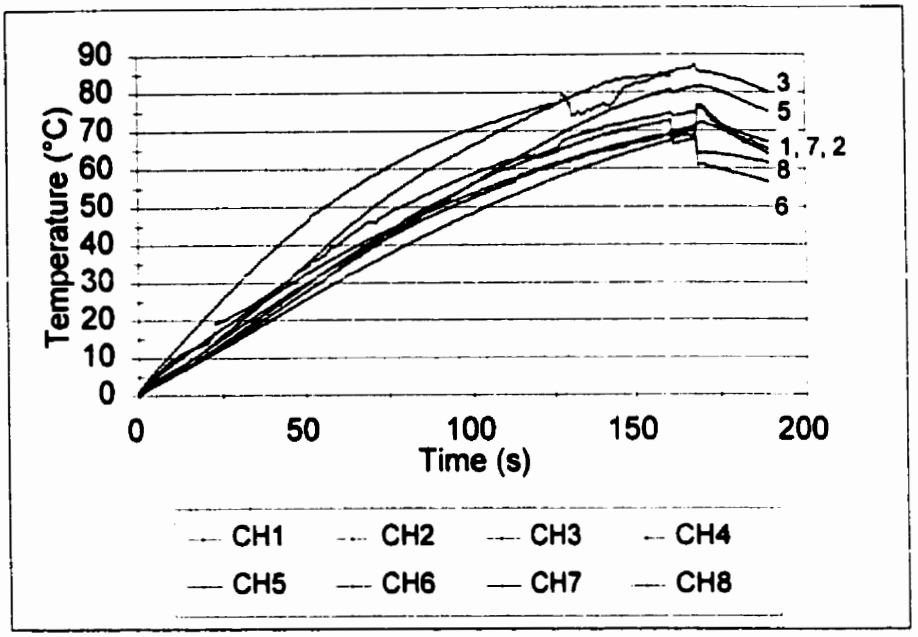
APPENDIX D WATER TEMPERATURE HISTORY



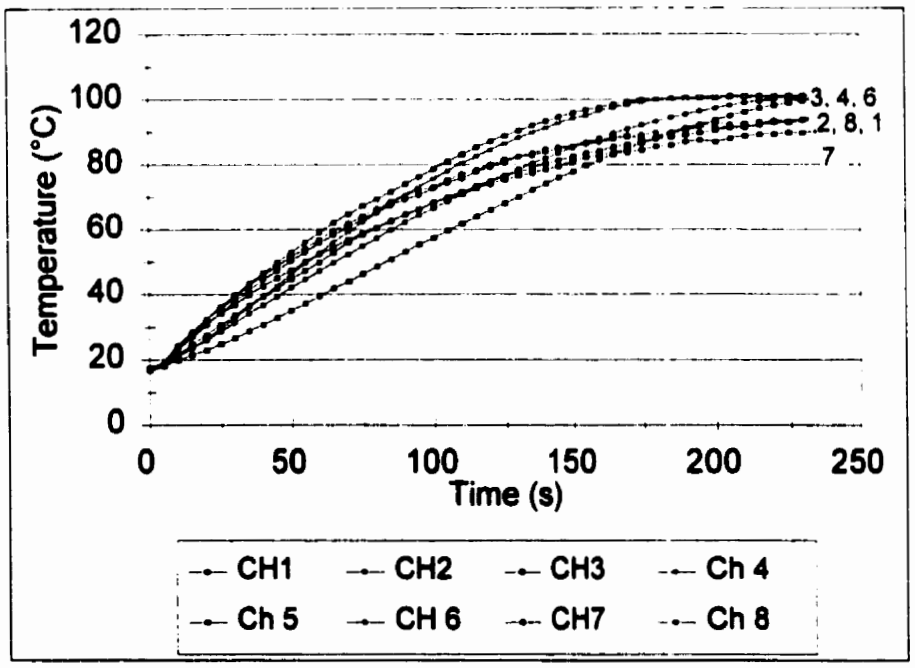
a) 6 cm



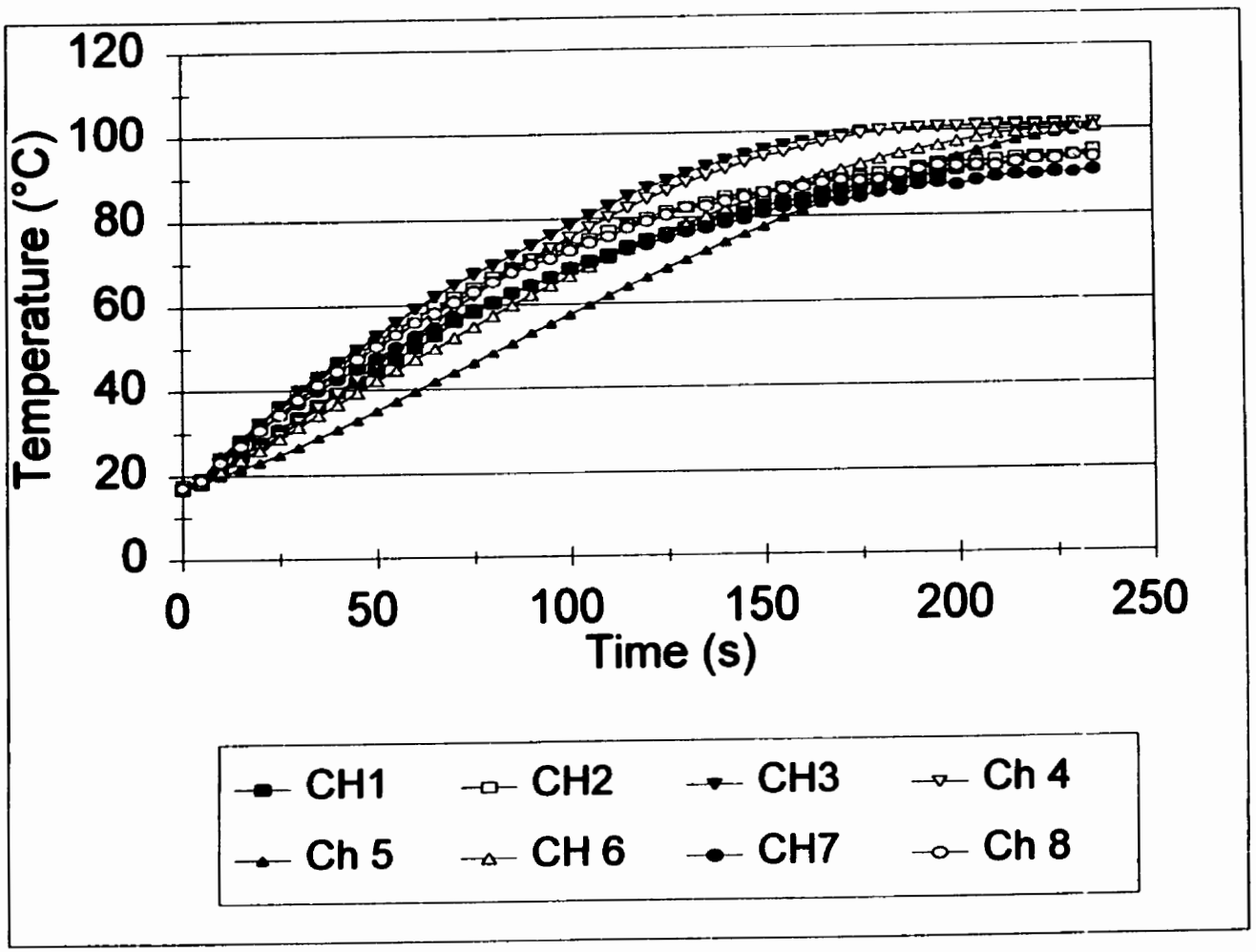
b) 8 cm


Temperature (°C)
90
80
70
60
50
40
30
20
10
0
0
50
100
150
200
Time (s)
3
5
1, 7, 2
8
6
CH1
CH2
CH3
CH4
CH5
CH6
CH7
CH8

c) 10 cm

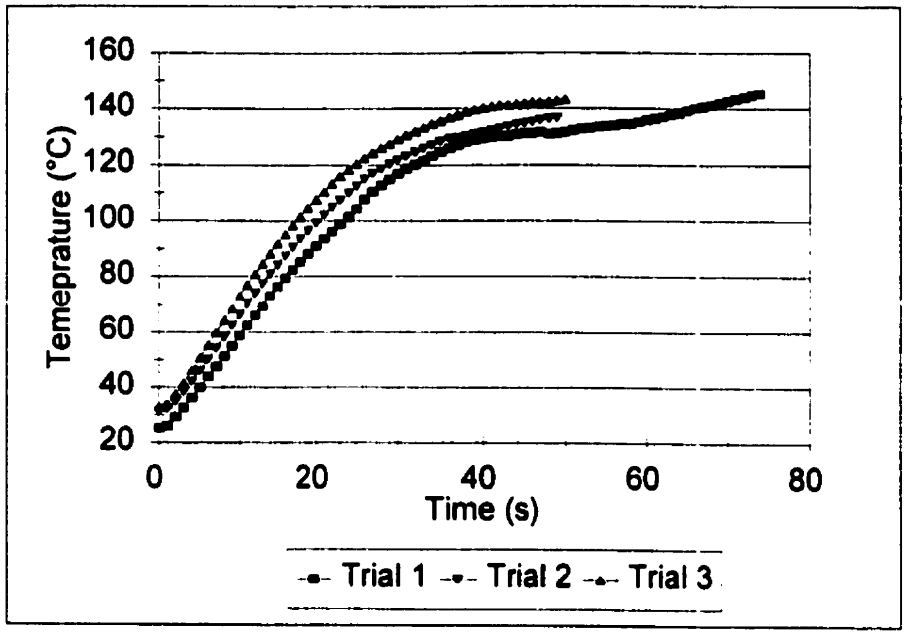

Temperature (°C)
120
100
80
60
40
20
0
0
50
100
150
200
250
Time (s)
3, 4, 6
2, 8, 1
7
CH1
CH2
CH3
Ch 4
Ch 5
CH 6
CH7
Ch 8

d) 12 cm

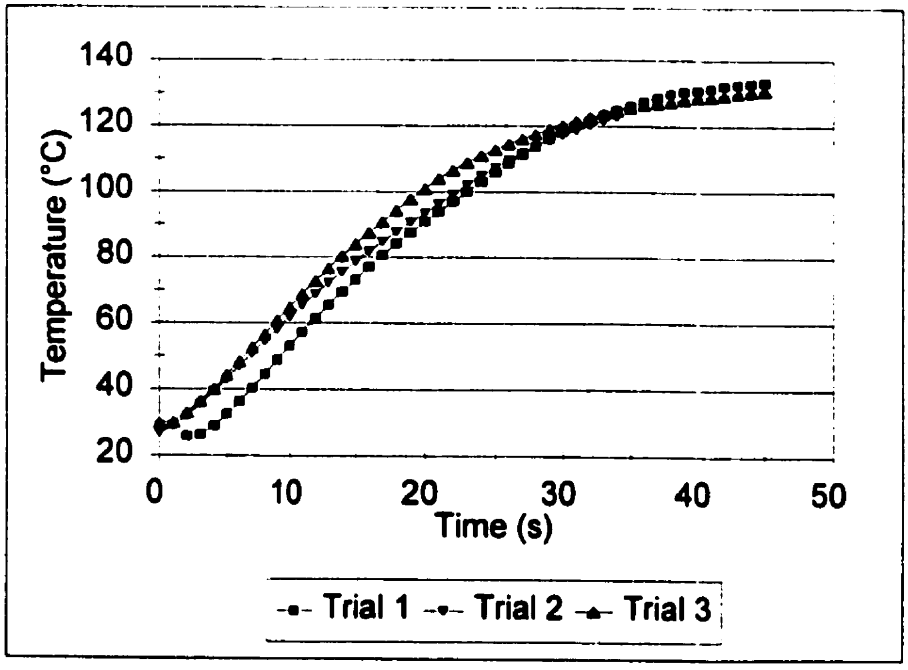


e) Comparison of temperatures across the width of the IR lamp (12 cm)

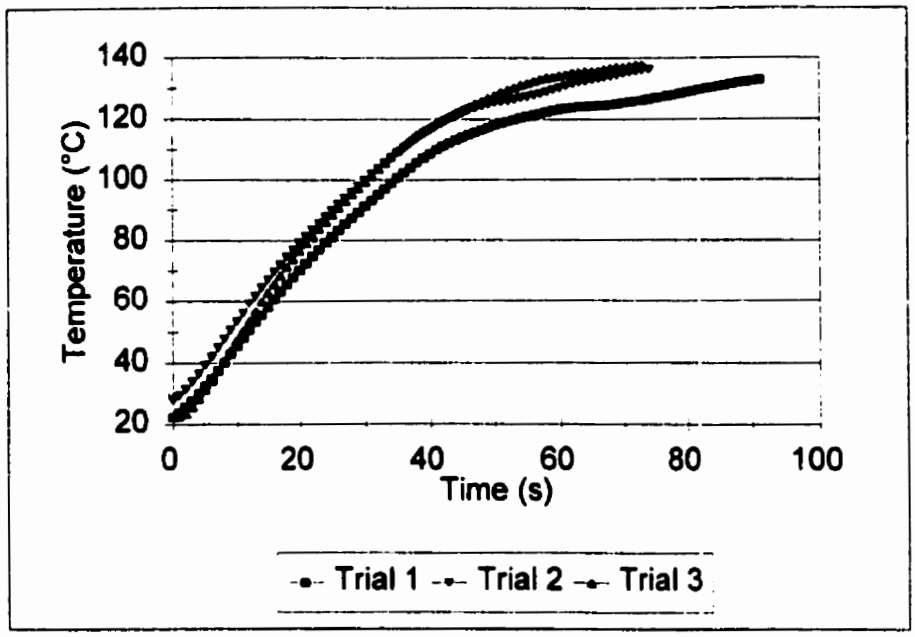
APPENDIX E TEMPERATURE OF DEHULLED YELLOW PEAS DURING MICRONIZATION



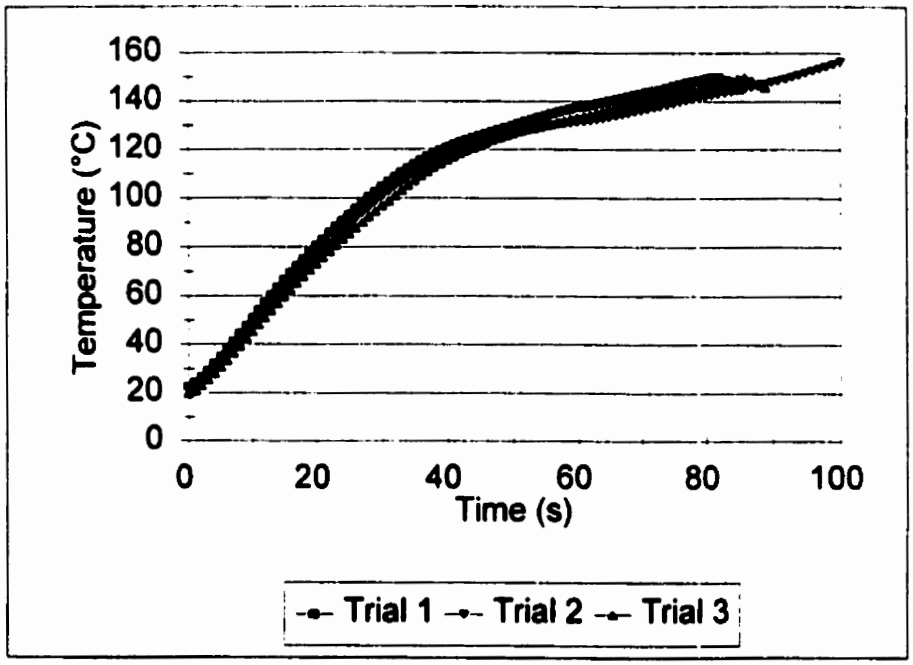
a) 23.2 kW/m², 26% mc



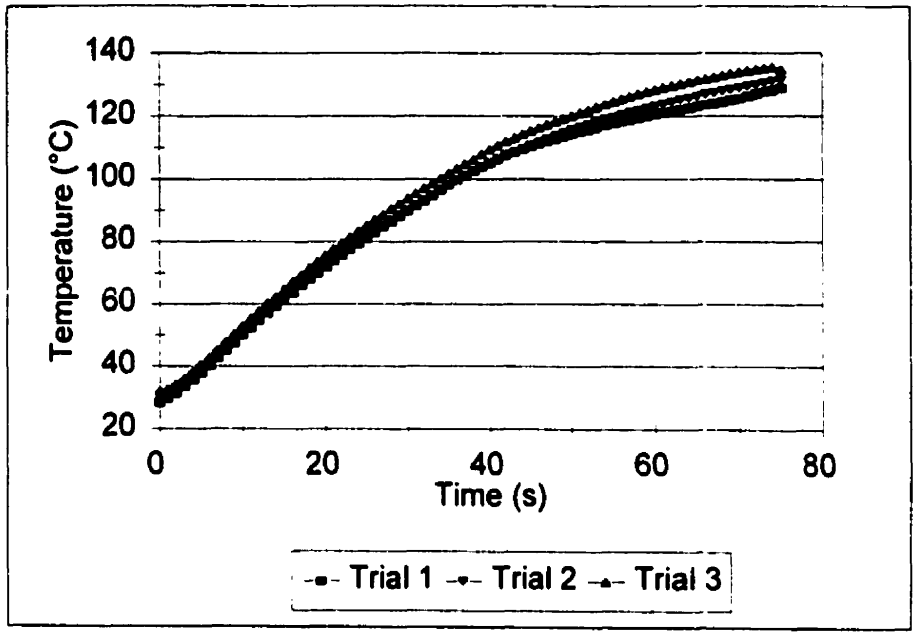
b) 23.2 kW/m², 28% mc



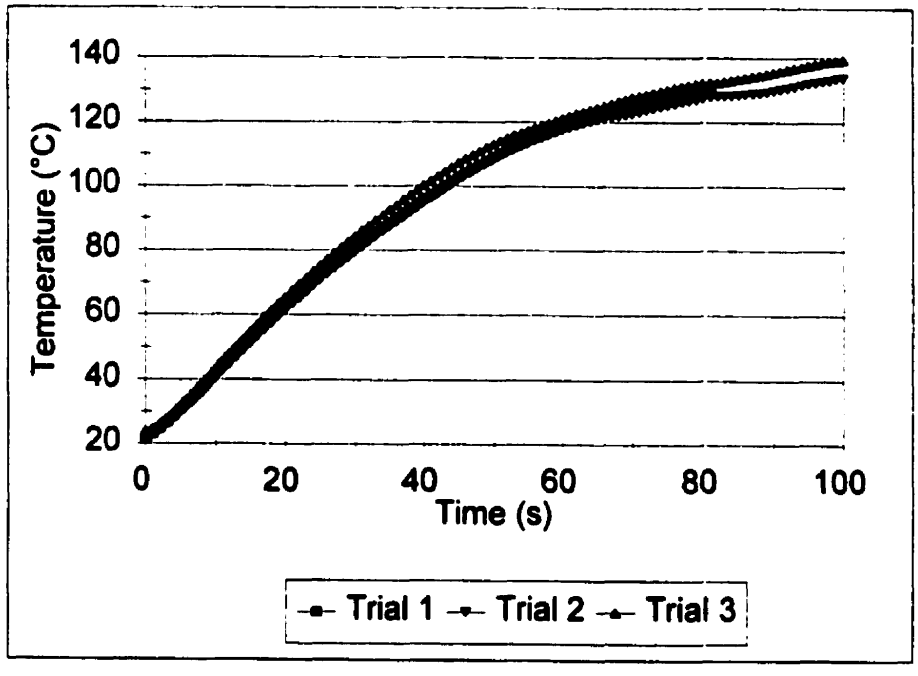
c) 16.8 kW/m², 26% mc



d) 16.8 kW/m², 28% mc

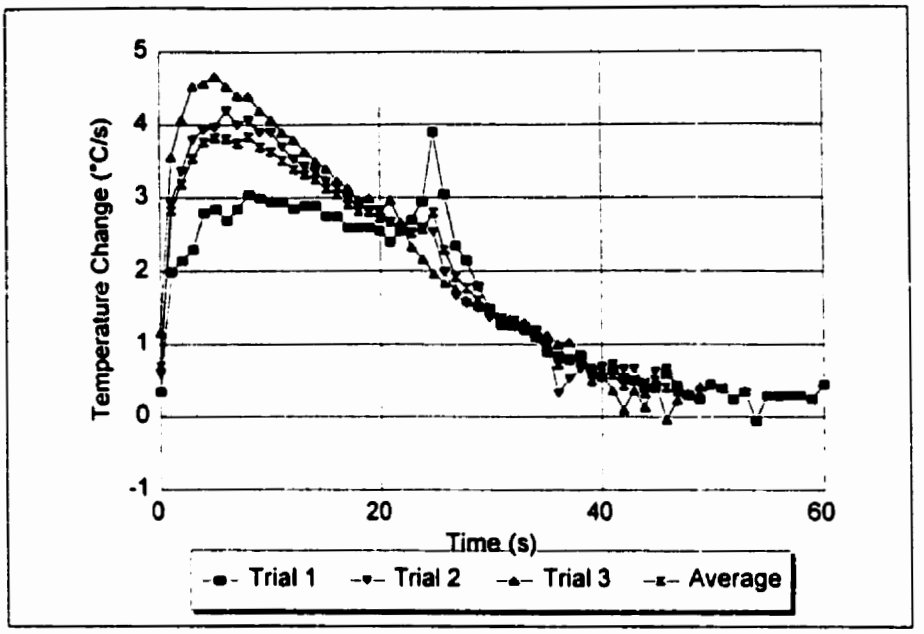


e) 12.3 kW/m², 26% mc

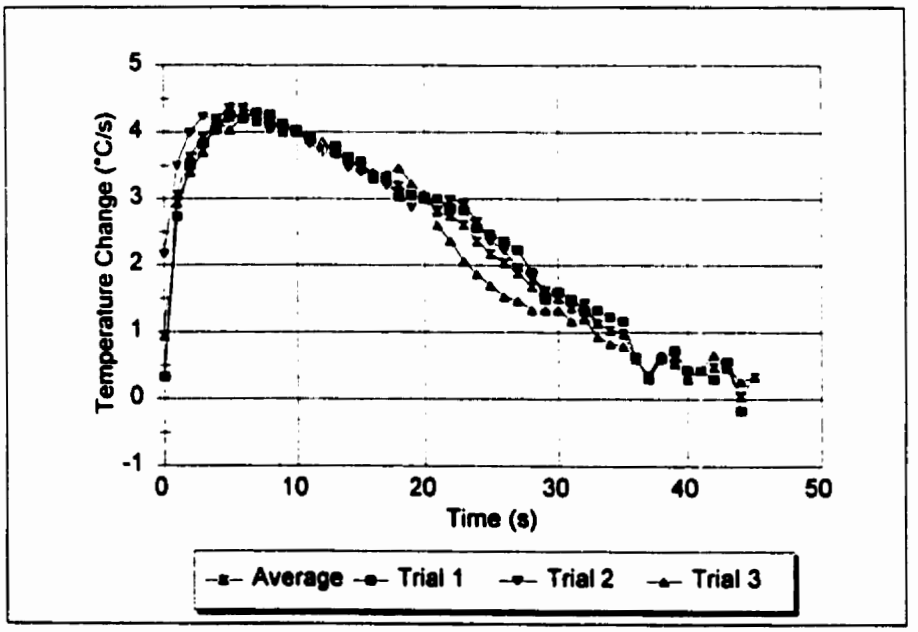


f) 12.3 kW/m², 28% mc

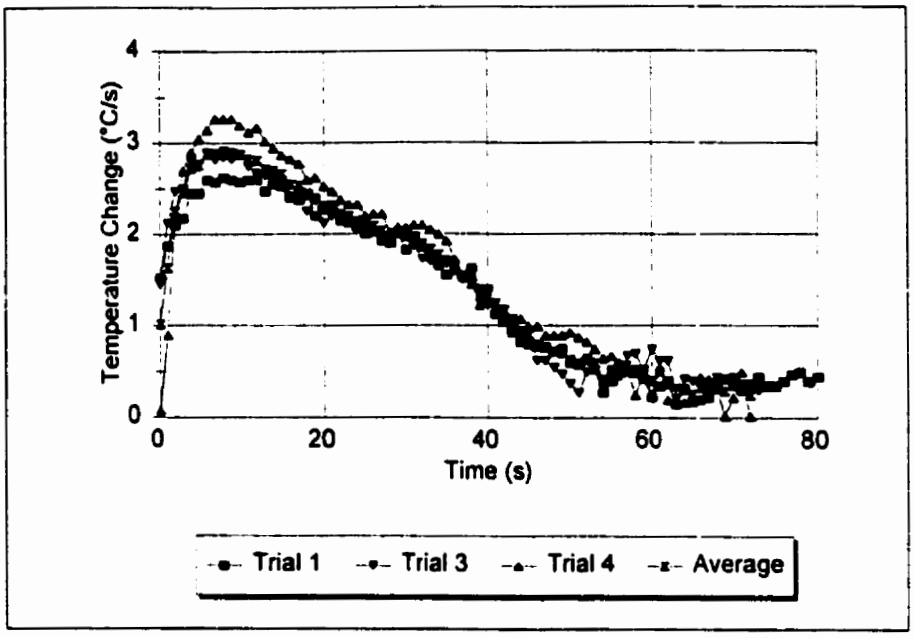
APPENDIX F TEMPERATURE CHANGE PER SECOND OF DEHULLED YELLOW PEAS



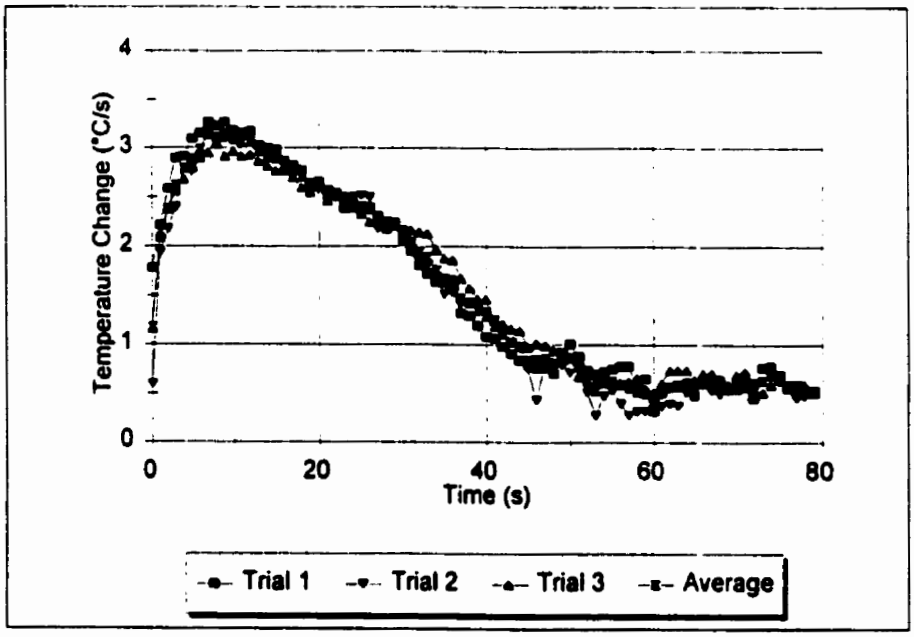
a) 23.2 kW/m², 26% mc



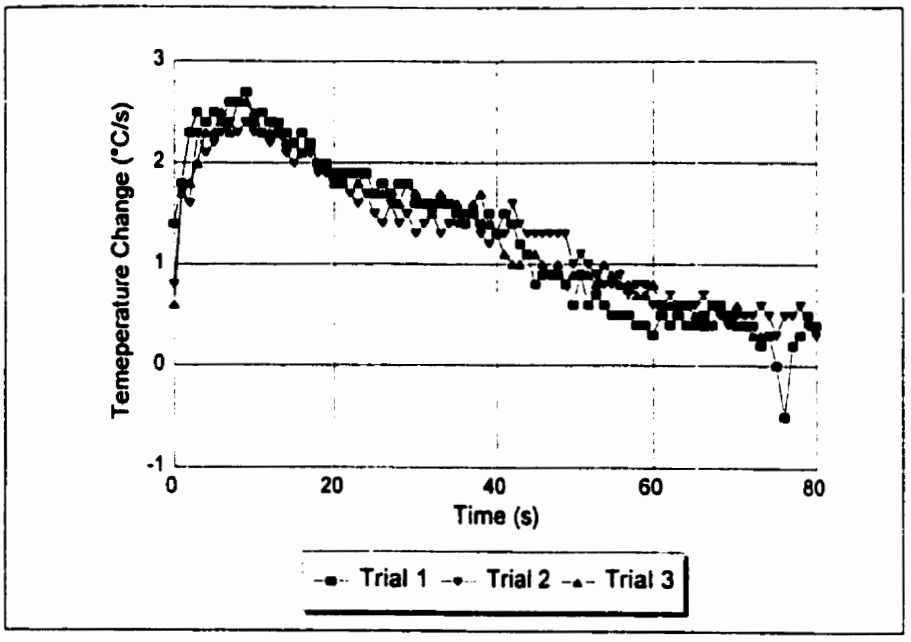
b) 23.2 kW/m², 28% mc



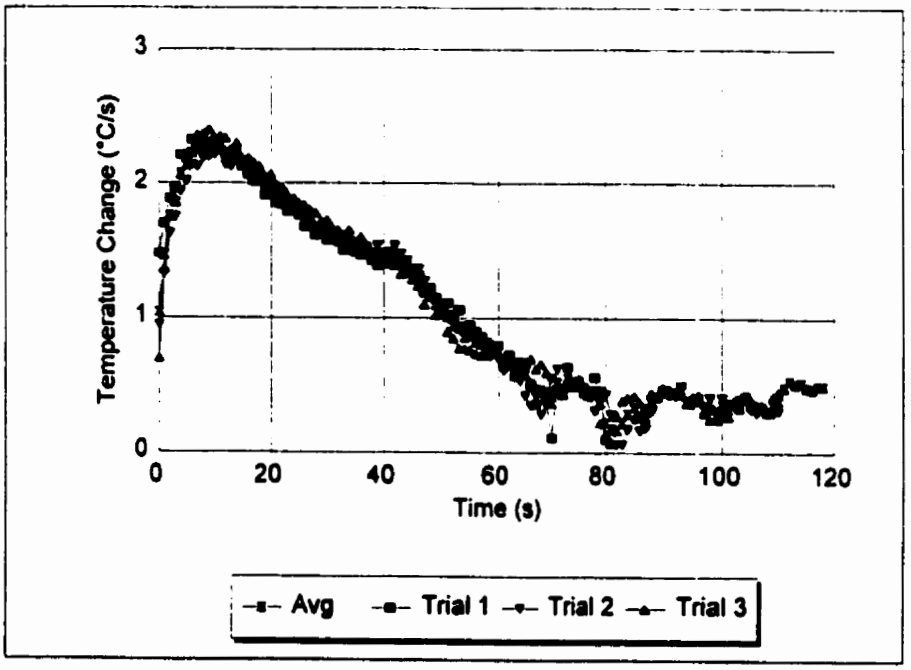
c) 16.8 kW/m², 26% mc



d) 16.8 kW/m², 28% mc

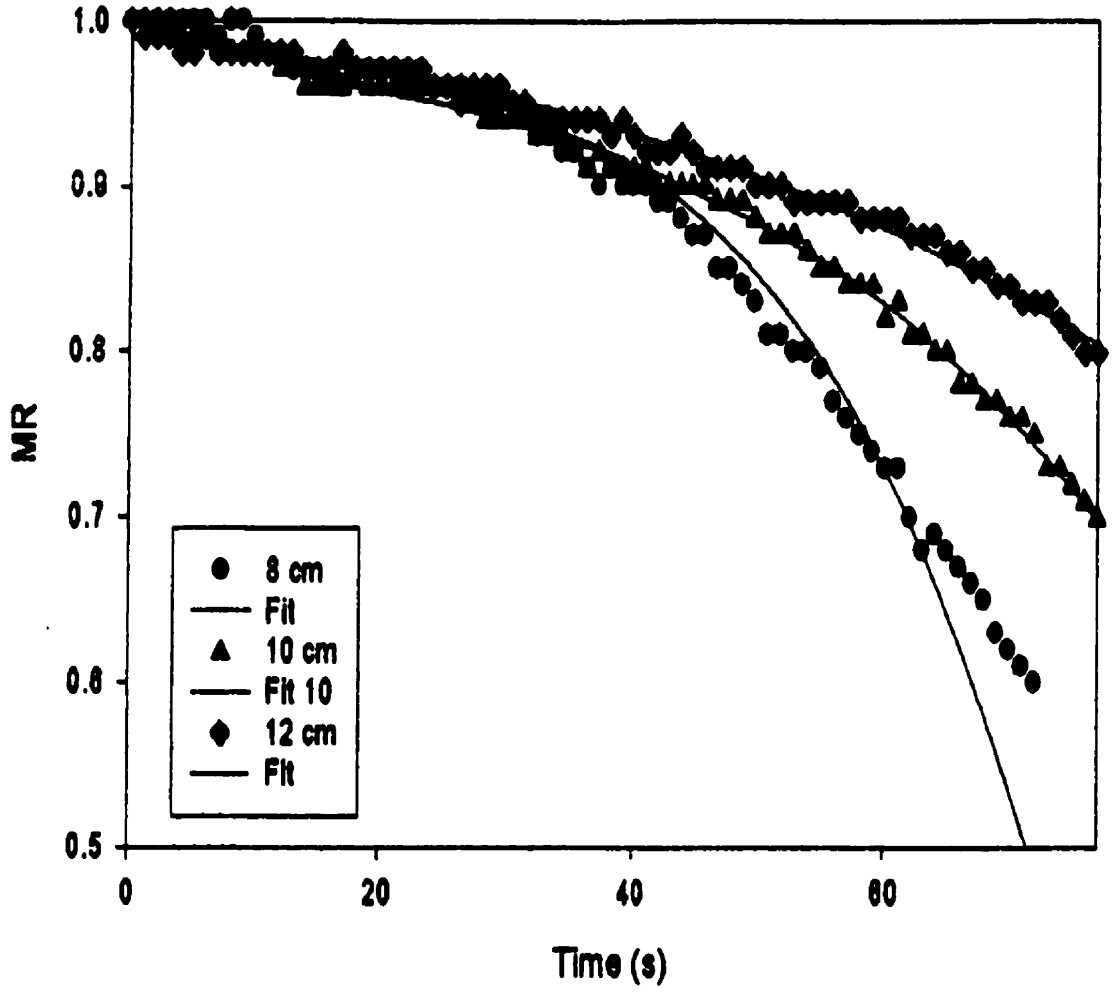


e) 12.3 kW/m², 26% mc

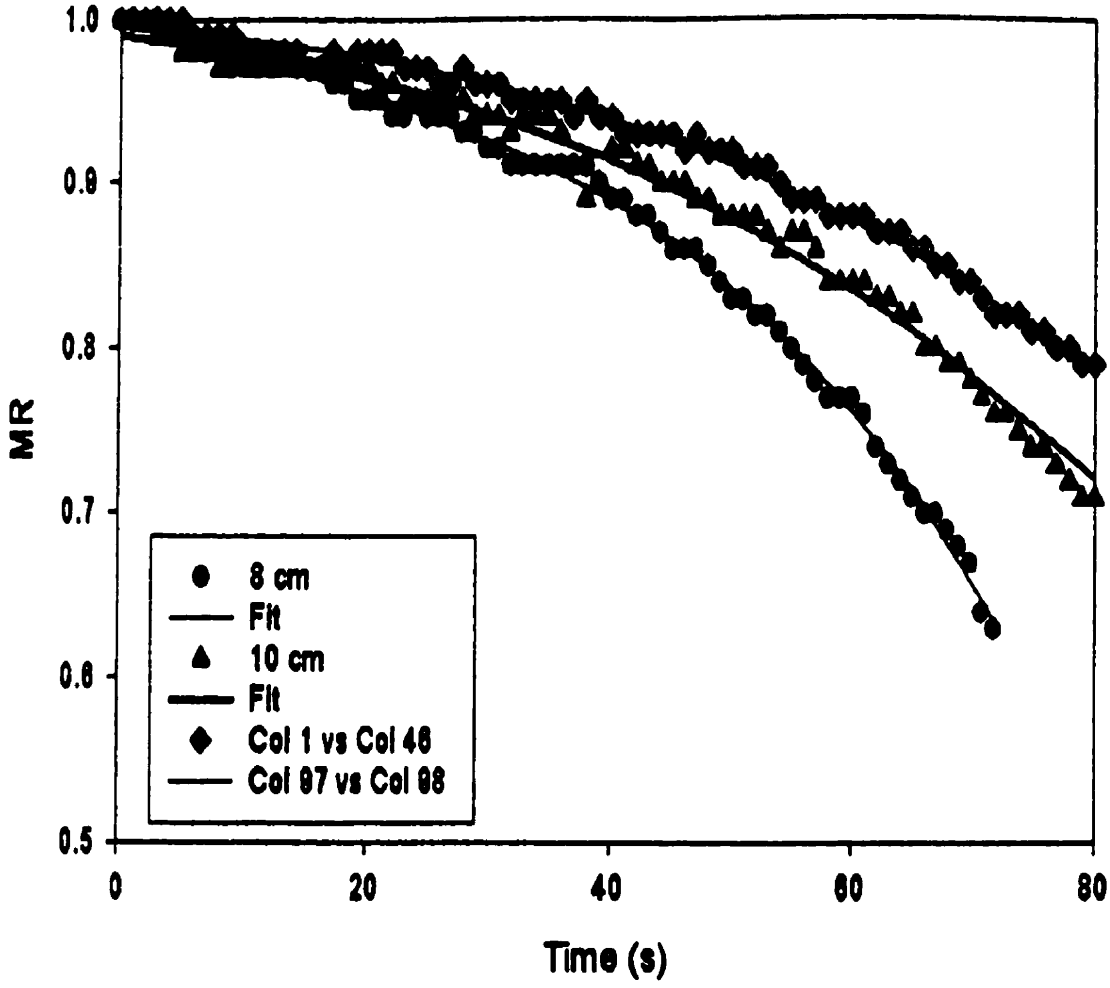


f) 12.3 kW/m², 28% mc

APPENDIX G FITTING MOISTURE RATIOS CURVES



MR Histories for different lamp heights at 26% original moisture content



MR Histories for different lamp heights at 28% original moisture content

APPENDIX H DATA SHEET FOR DIGESTIBILITY EXPERIMENTS

Digestible Dry Matter

Treatment	Sample Weight (g)	Pepsin Weight (g)	Dry Matter after Dialysis (g)	Digested Dry Matter (%)
Reference 1	5.1509	0.5085	2.8167	45.32
Reference 2	5.0420	0.5024	– [a]	– [a]
16.8 kW/m^2; 1 [b]	4.9543	0.5016	1.4573	70.59
16.8 kW/m^2; 2 [b]	4.9598	0.5036	1.3830	72.12
23.2 kW/m^2; 1 [b]	5.0270	0.5006	1.4495	71.17
23.2 kW/m^2; 2 [b]	5.0839	0.5049	1.3149	74.14

[a] Tube broke during dialysis/digestion
[b] Initially at 26% mc when micronized

$$\text{Dry Matter Digestibility} = \frac{(M_s - M_{DM})}{M_s} \cdot 100\%$$

where:
M_s = Sample Weight (g)
M_{DM} = Dry Matter Weight (g)

Protein Remaining After Digestion

Treatment	Sample Weight (g)	Volume of Acid (ml)	Remaining Protein (%)	Average Remaining Protein (%)	Standard Deviation (%)
Reference 1	0.5017	3.753	6.62	6.79	0.18
	0.5062	3.988	6.97		
16.8 kW/m^2; 1[b]	0.5028	5.364	9.43	9.52	0.08
	0.5037	5.466	9.60		
16.8 kW/m^2; 2[b]	0.5065	4.637	8.10	7.75	0.34
	0.5038	4.223	7.41		
23.2 kW/m^2; 1[b]	0.5026	5.521	9.71	9.66	0.06
	0.5049	5.482	9.60		
23.2 kW/m^2; 2[b]	0.5107	5.865	10.16	9.41	0.75
	0.5030	4.926	8.66		
Undigested reference	0.5184	12.49	21.31	21.15	0.24
	0.5096	12.29	21.33		
	0.5113	12.03	20.81		

[b] Initially at 26% mc when micronized

$$\% \text{ Protein} = \frac{N * N[HCl] * V * 6.25}{M_s} * 100\%$$

where:

N	=	Atomic weight of N, 14.01g/mol
N[HCl]	=	Normality of HCl, moles/L
V	=	Volume of acid, L
6.25	=	Protein conversion factor
M_s	=	Sample weight, g

Digestible Protein

Sample	Protein Left after Digestion (%)	Original Protein (g)	Protein Digested (g)	Protein Digested (%)
Reference 1	6.79	1.0893	0.1913	82.44
Reference 2	–	–	–	–
16.8 kW/m^2; 1 [b]	9.52	1.0477	0.1387	86.76
16.8 kW/m^2; 2 [b]	7.75	1.0489	0.1072	89.78
23.2 kW/m^2; 1 [b]	9.66	1.0631	0.1400	86.83
23.2 kW/m^2; 2 [b]	9.41	1.0751	0.1237	88.49
Undigested reference	21.15			

Original Protein = M_s*%P

where:
M_s = Sample weight, g
%P = Percent protein in undigested reference sample, 21.15 g

Weight of Protein Digested = M_{DM}*%P

where:
M_{DM} = Dry Matter weight, g

% Protein Digested = [(Original Protein) - (Protein Digested)]/(Original Protein)

APPENDIX I DSC7 MELTING PROFILES FOR SELECTED GRAPHS

Reference Sample: Trial 3
Temperature Range: 50-80°C
Temperature Range: 70-110°C

23.2 kW/m²; 26% mc; Trial 1
Temperature Range: 40-70°C
Temperature Range: 70-110°C

23.2 kW/m²; 28% mc; Trial 3
Temperature Range: 40-70°C

16.8 kW/m²; 26% mc; Trial 3
DSC performed immediately after processing
Temperature Range: 45-70°C

16.8 kW/m²; 26% mc; Trial 1
DSC performed 24h after processing
Temperature Range: 45-70°C
Temperature Range: 70-110°C

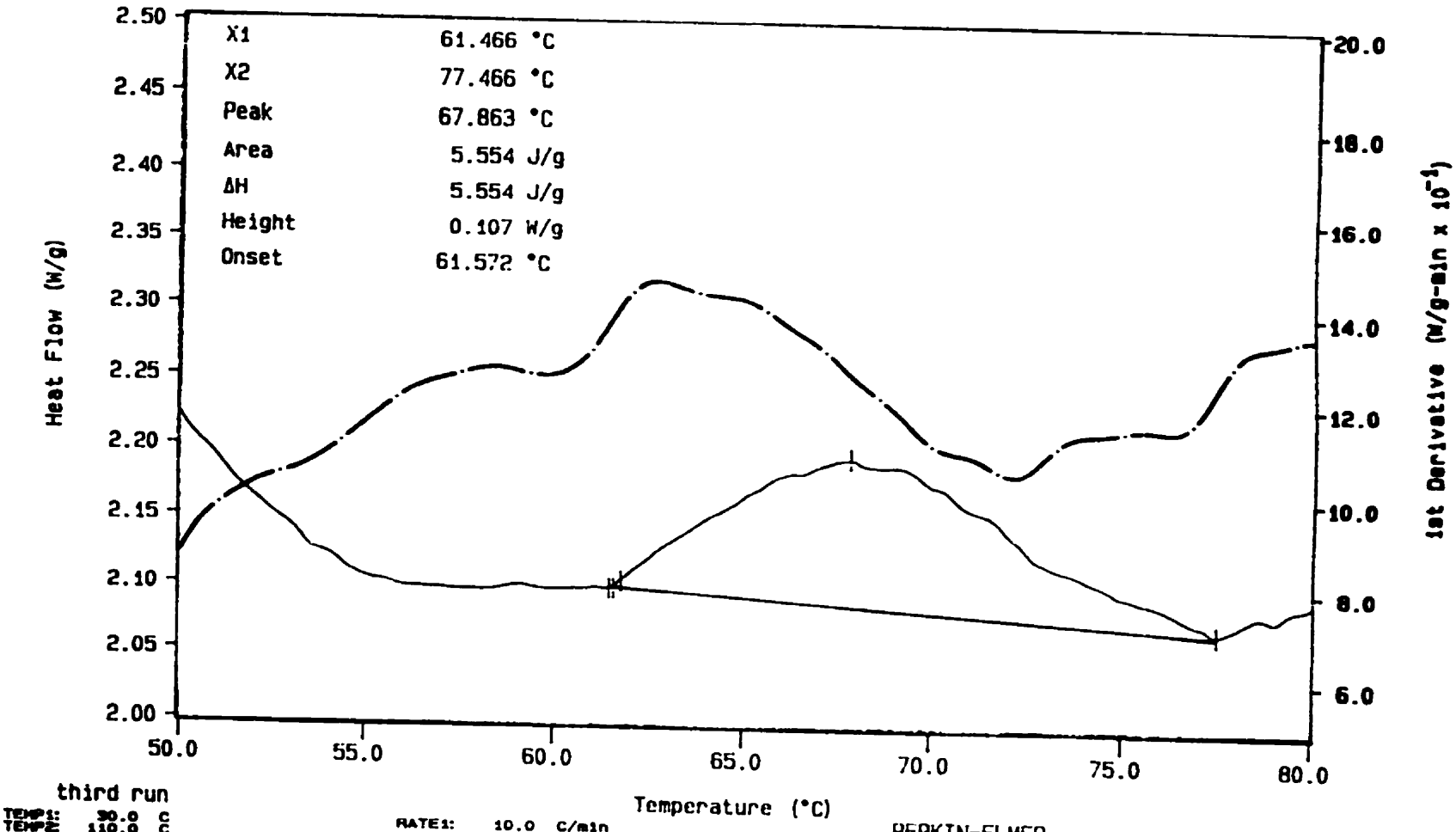
16.8 kW/m²; 26% mc; Trial 2;
DSC performed 2 wk after processing
Temperature Range: 40-70°C
Temperature Range: 70-110°C

16.8 kW/m²; 28% mc; Trial 2;
Temperature Range: 40-70°C

12.3 kW/m²; 26% mc; Trial 3
Temperature Range: 40-70°C
Temperature Range: 70-110°C

12.3 kW/m²; 28% mc; Trial 2
Temperature Range: 59-66°C
Temperature Range: 92-99°C

File info: pearef3 Mon May 3 15:43:32 1999
Sample Weight: 7.780 mg
non micronized non moisturized



third run
TEMP1: 30.0 C
TEMP2: 110.0 C
RATE1: 10.0 C/min

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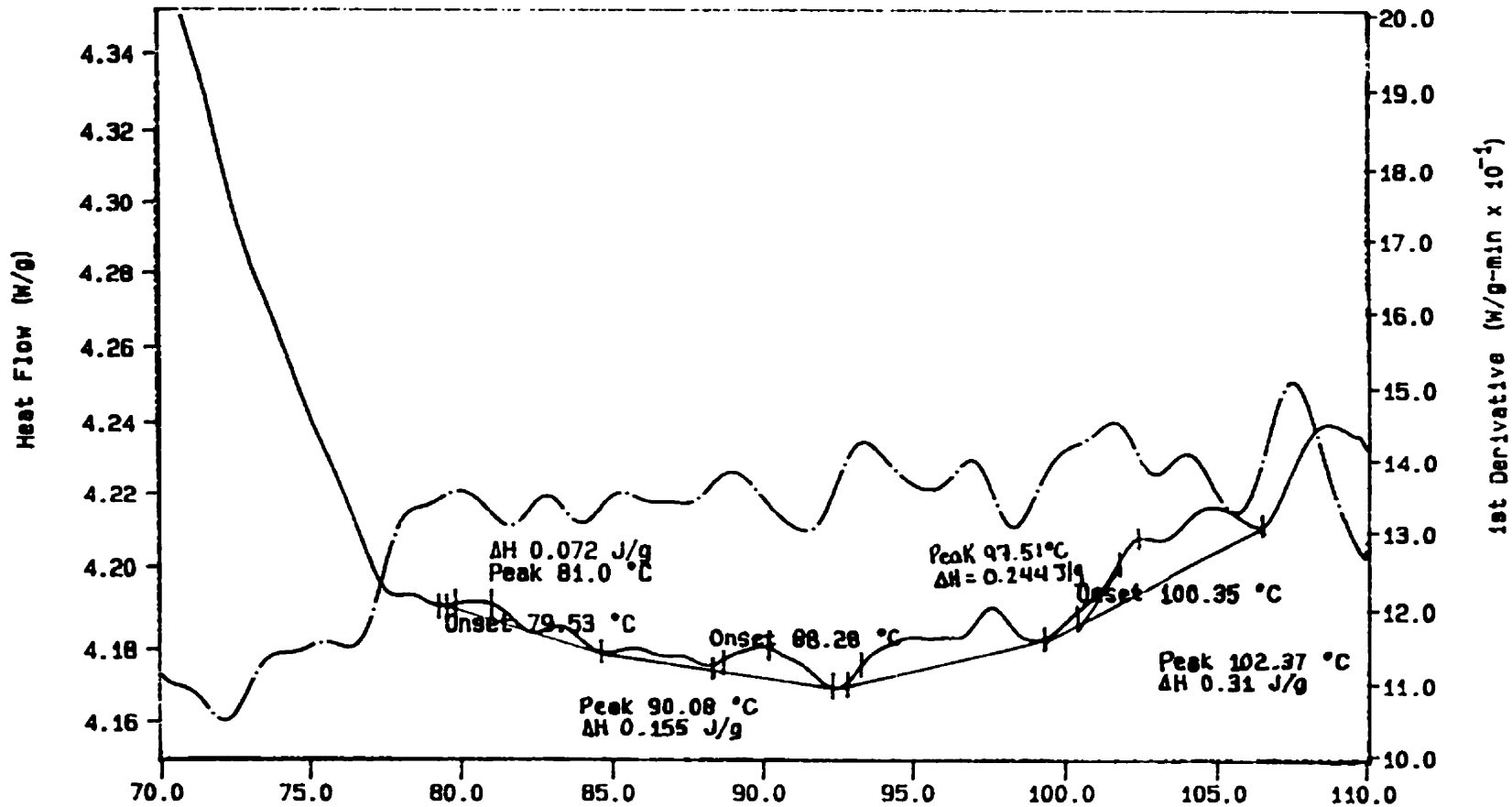
Treatment: Reference Sample : Trial 3

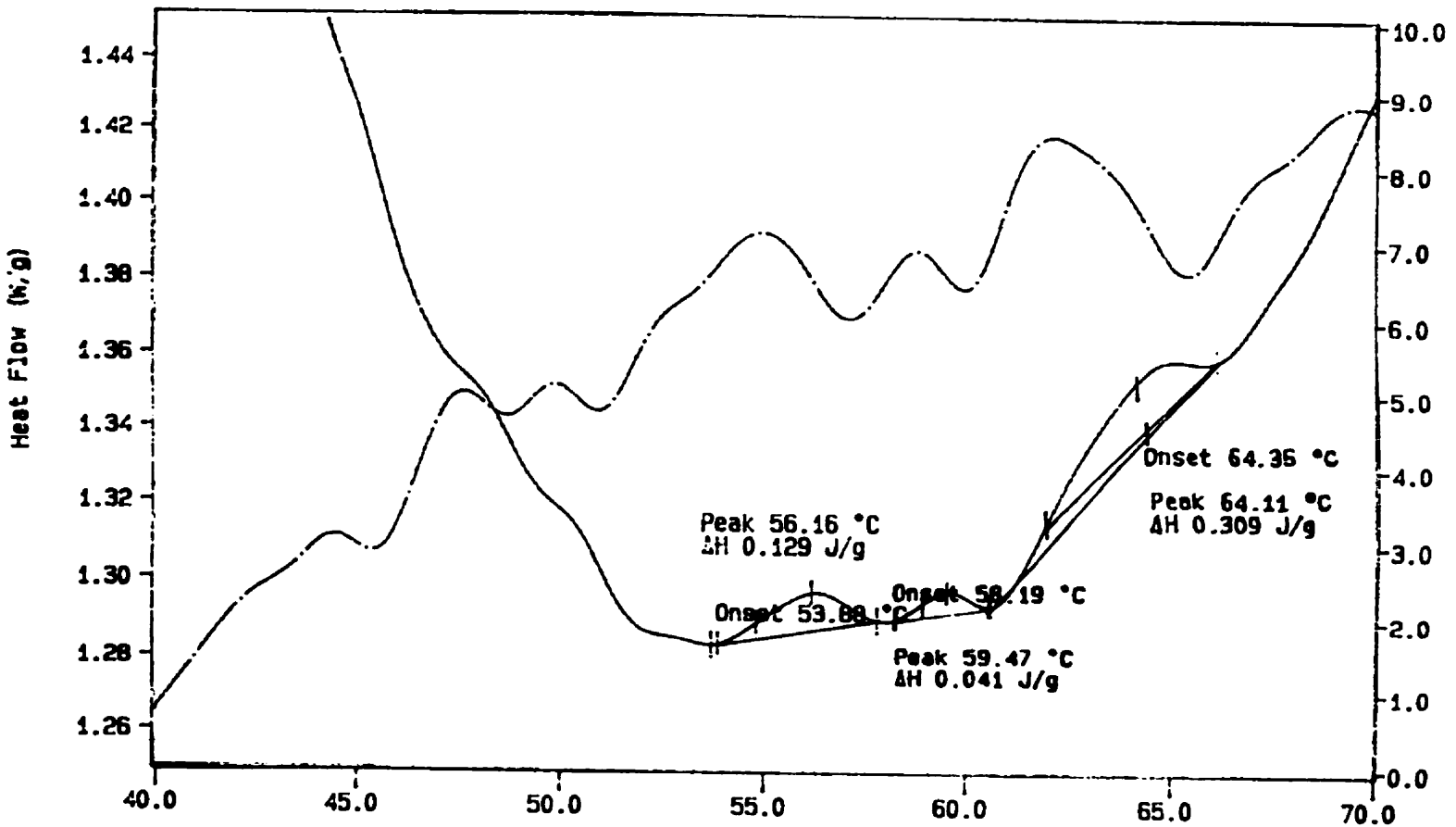
File info: pearef3 Mon May 3 15:43:32 1999
Sample Weight: 7.780 mg
non micronized non moisturized



third run
TEMP1: 30.0 C
TEMP2: 110.0 C
RATE1: 10.0 C/min
PERKIN-ELMER
7 Series Thermal Analysis System

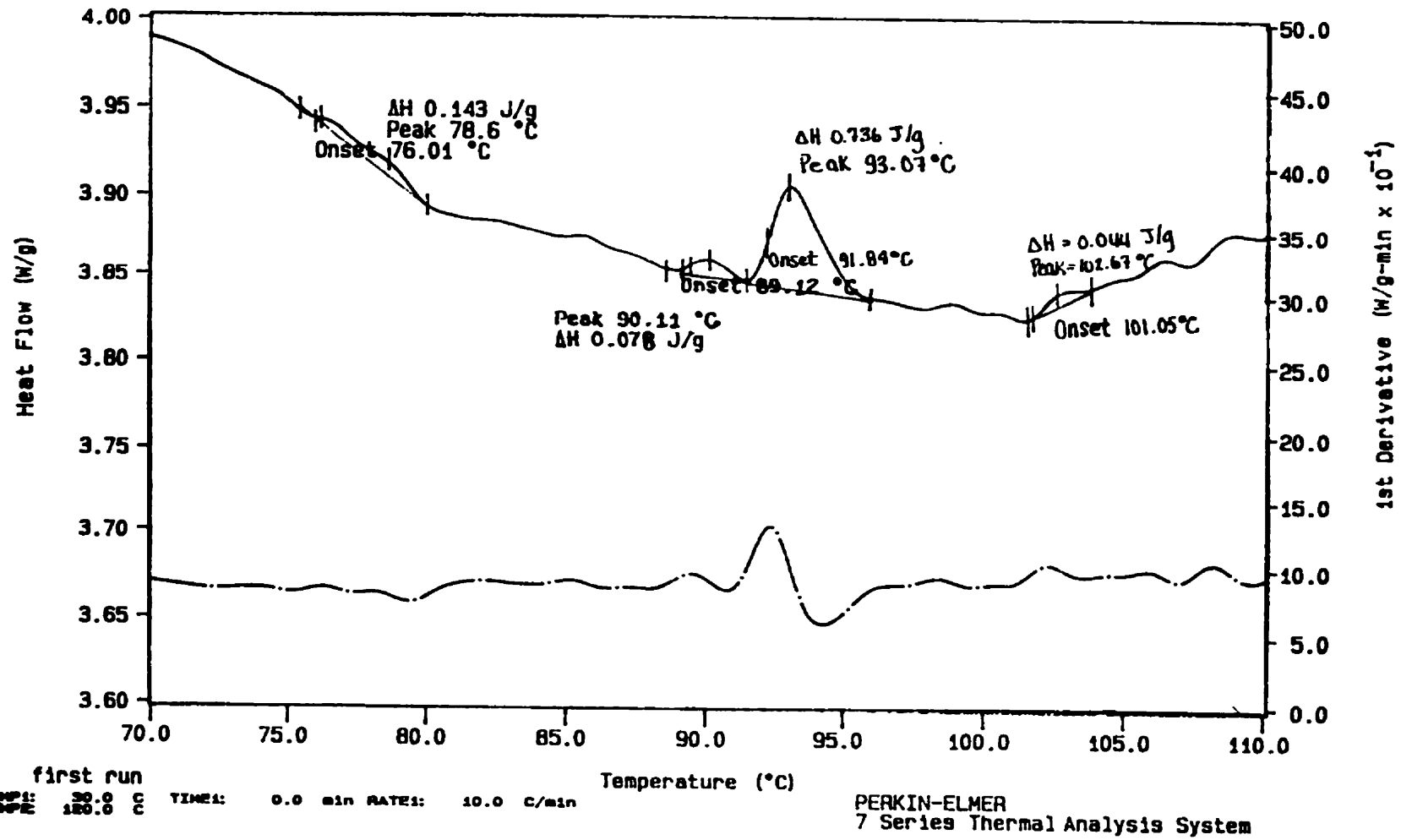
Treatment: Reference Sample: Trial3

File info: 828iarea1 Fri Apr 23 15:07:21 1999
Sample Weight: 8.920 mg



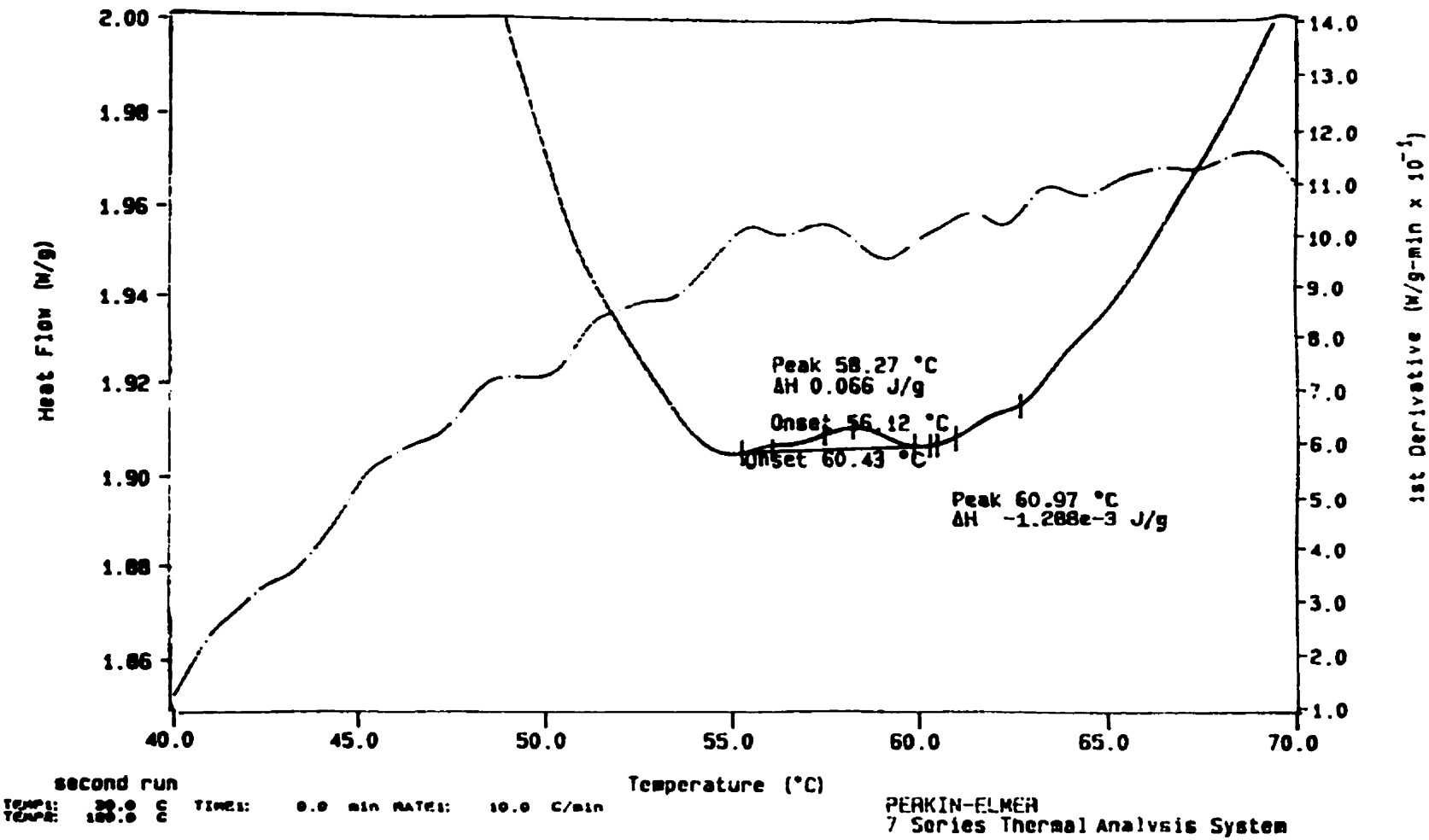
Treatment: 23.2 KW/m^2 : 26% mc. : Trial 1

File info: 8281areal Fri Apr 23 15:07:21 1999
Sample Weight: 8.920 mg



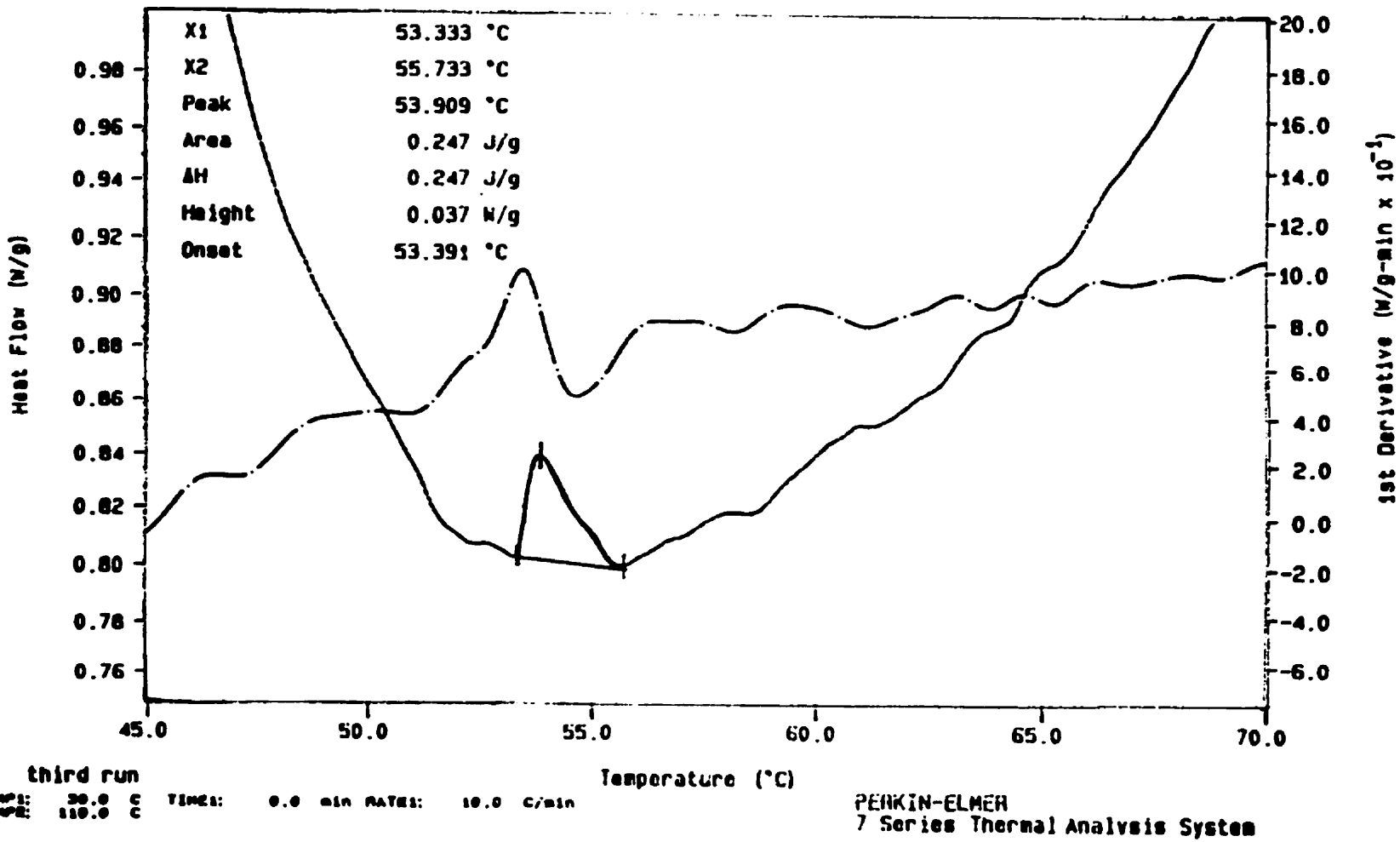
Treatment: 23.2 KW/m^2 ; 26%mc : Trial 1

File info: 8303breal Fri Apr 23 14:45:16 1999
Sample Weight: 9.470 mg



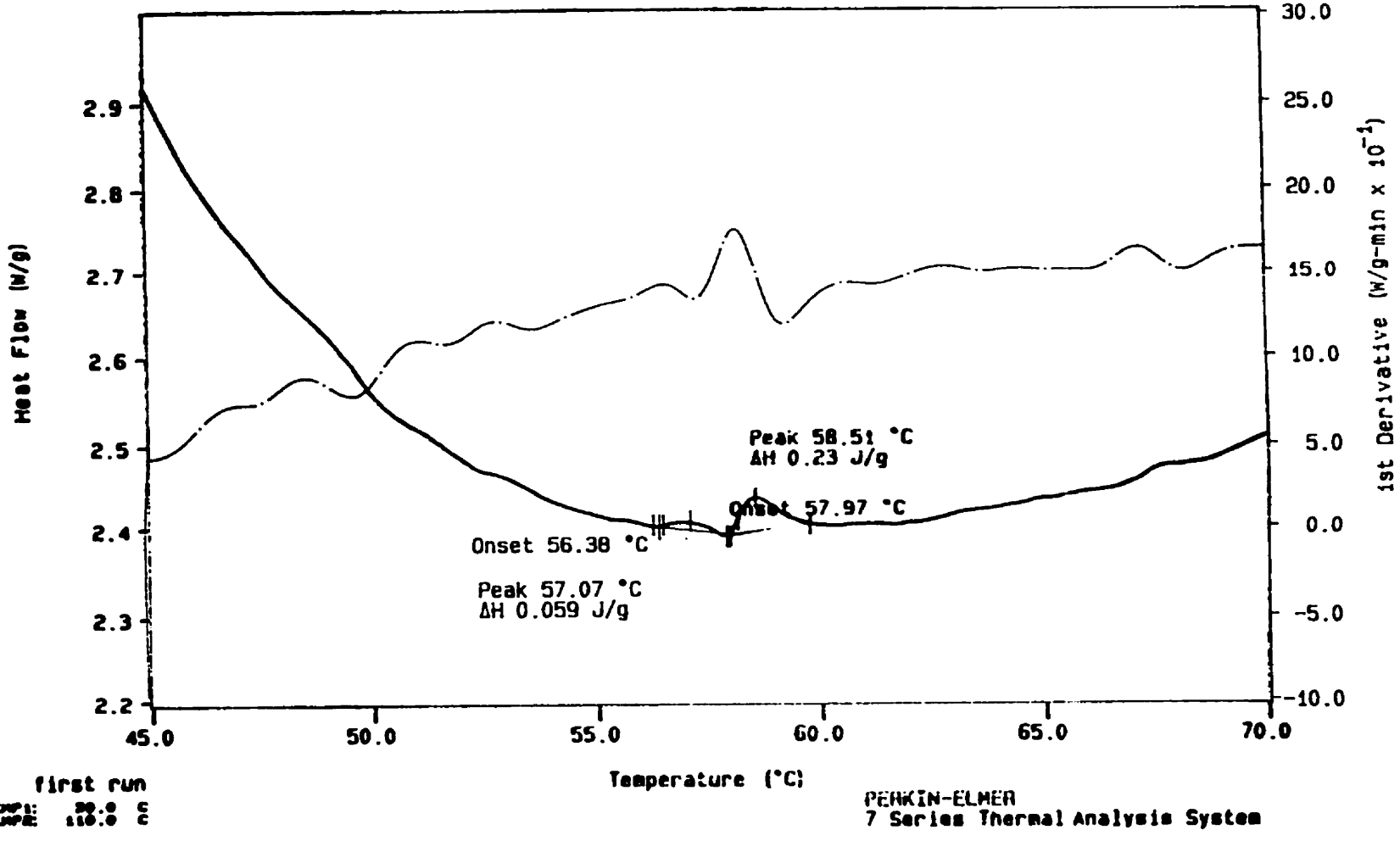
Treatment: 23.2 kW/m^2; 28%mc : Trial 3

File info: 1028asap3 Thu May 6 15:53:59 1999
Sample Weight: 9.620 mg



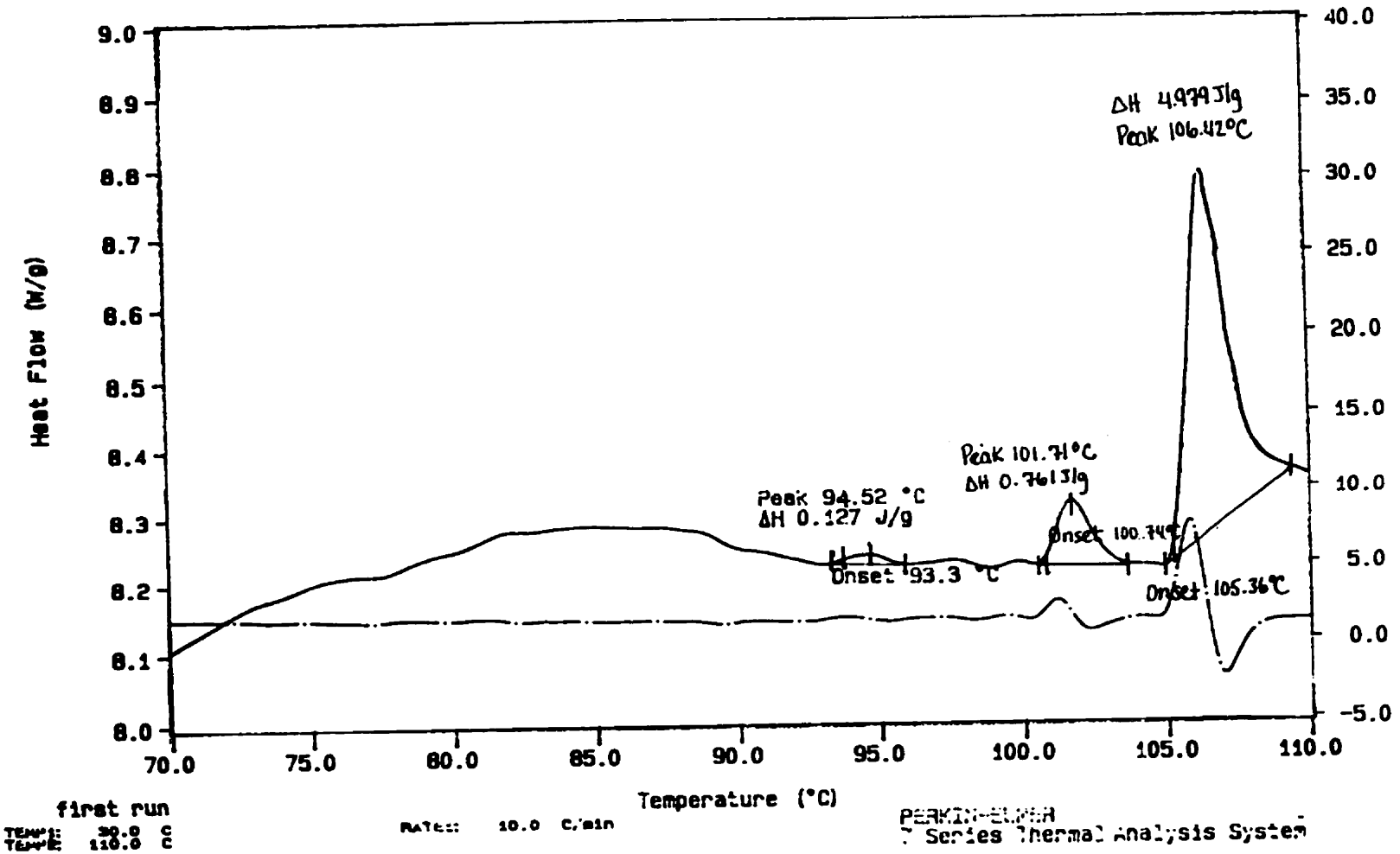
Treatment: 1.68 kW/m²; 26% mc; DSC 7 performed immediately after processing

File info: 102824h1 Fri May 7 15:41:57 1999
Sample Weight: 8.240 mg



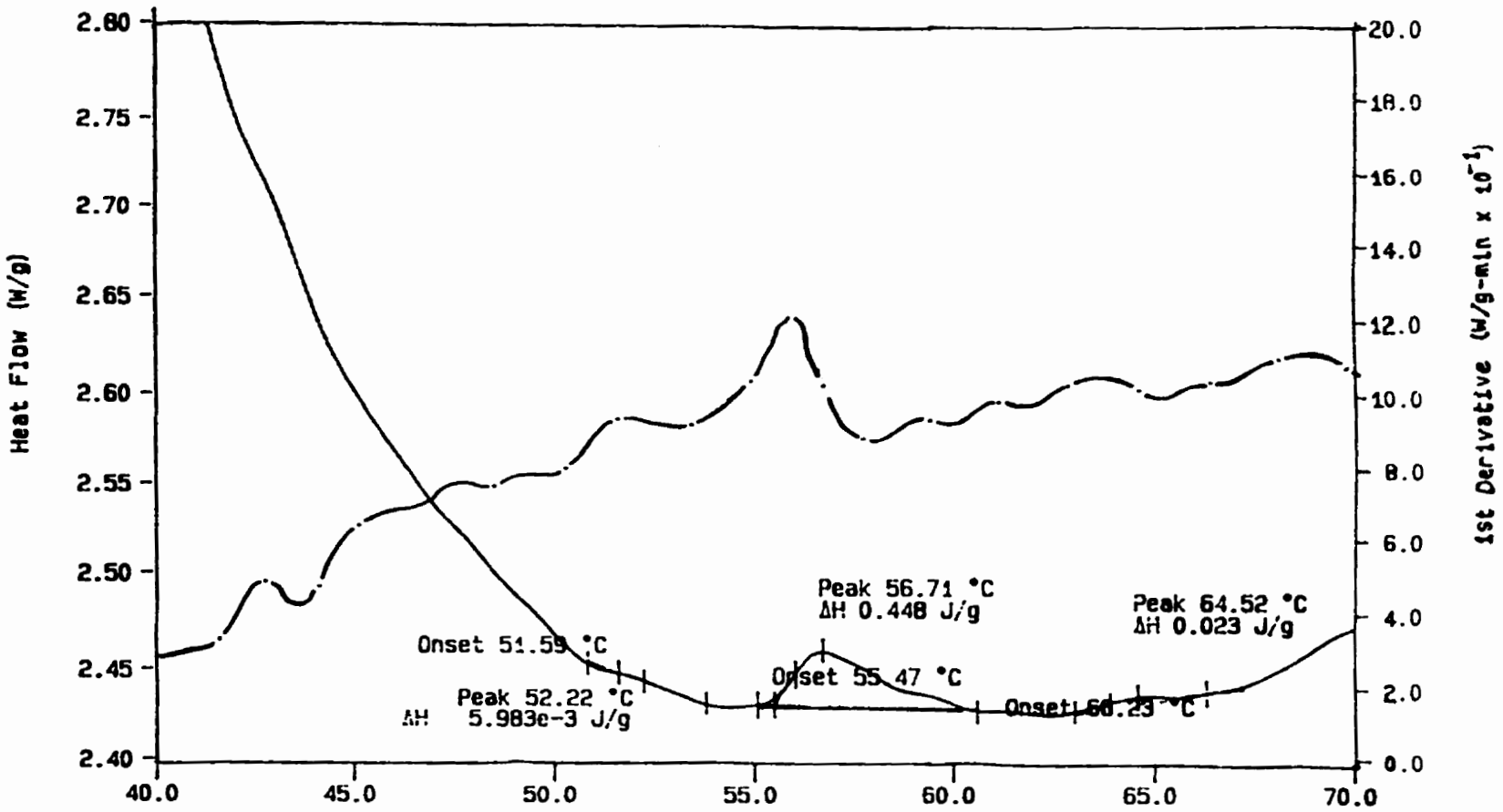
Treatment: 16.8 kW/m^2; 26%mc; DSC7 performed 24 hours after processing

File Info: 102824h1 Fri May 7 15:41:57 1999
Sample Weight: 8.240 mg



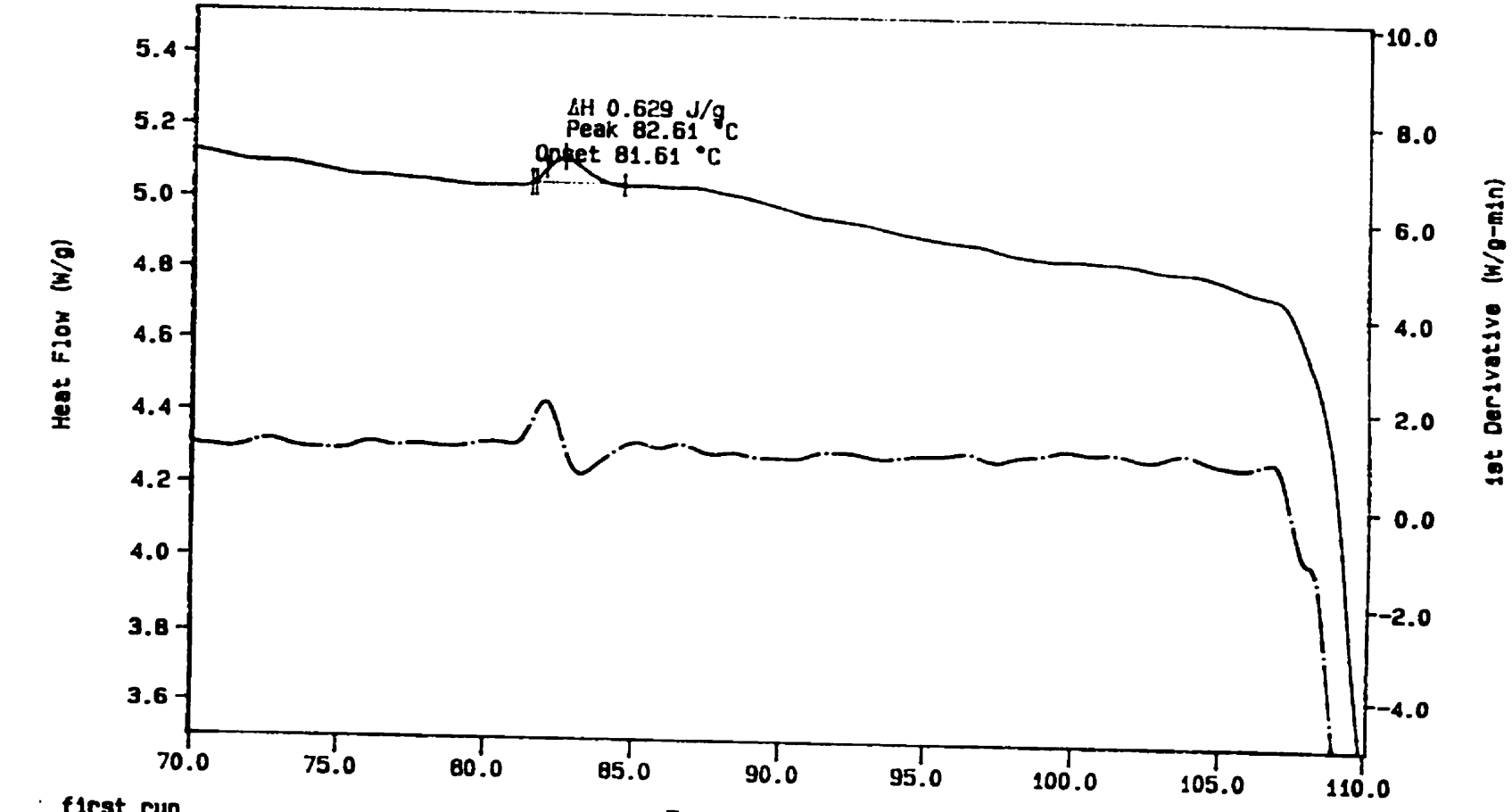
Treatment: 16.8kW/m²; 26%mc; Trial 1; DSC performed 24 hours after processing

File info: 10282a Thu Apr 22 14:46:06 1999
Sample Weight: 10.320 mg



Treatment: 16.8 kW/m²; 26%mc; Trial 2; 2wk after processing

File info: 10282a Thu Apr 22 14:46:06 1999
Sample Weight: 10.320 mg



first run
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 10.0 C/min
TEMP2: 110.0 C

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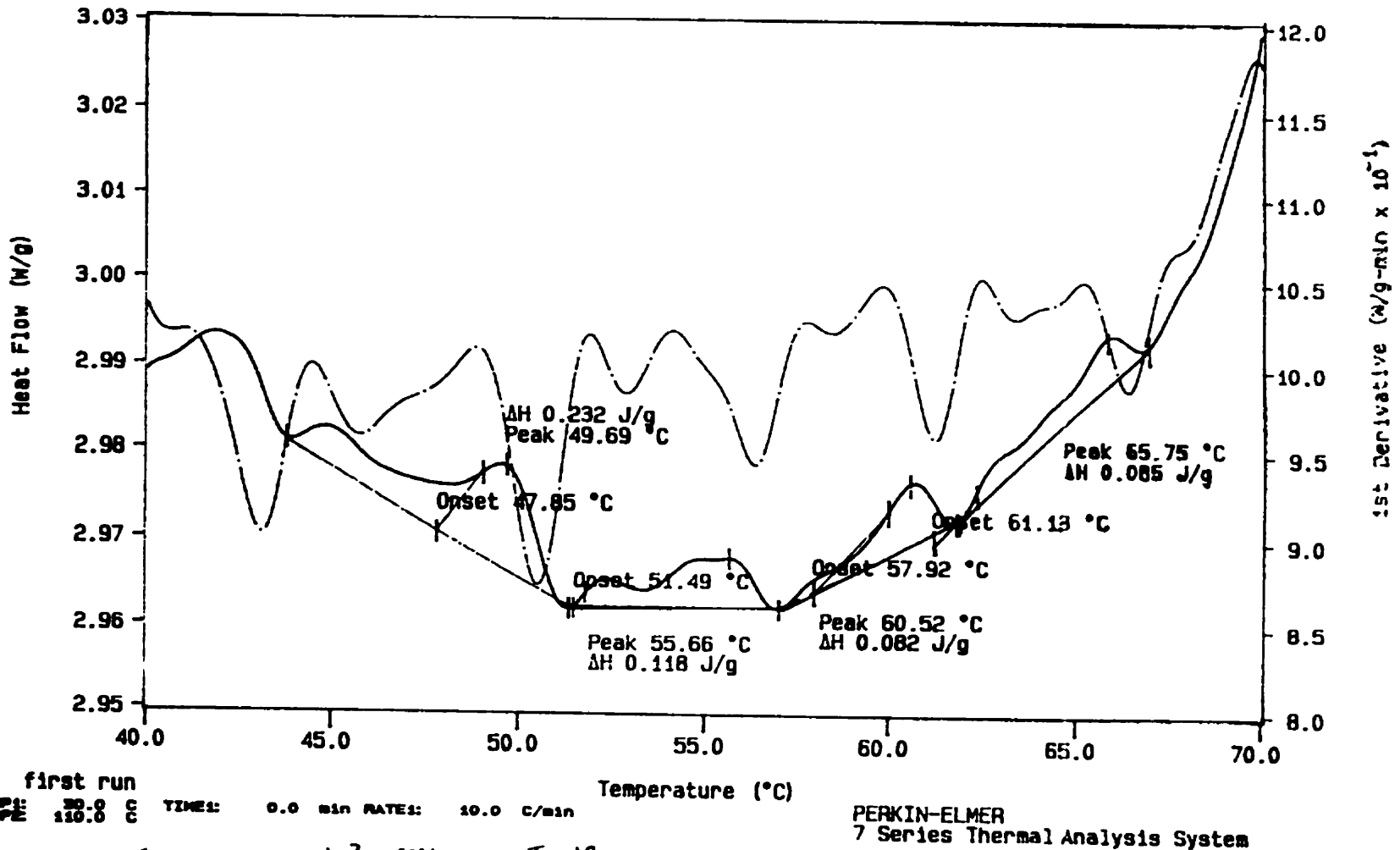
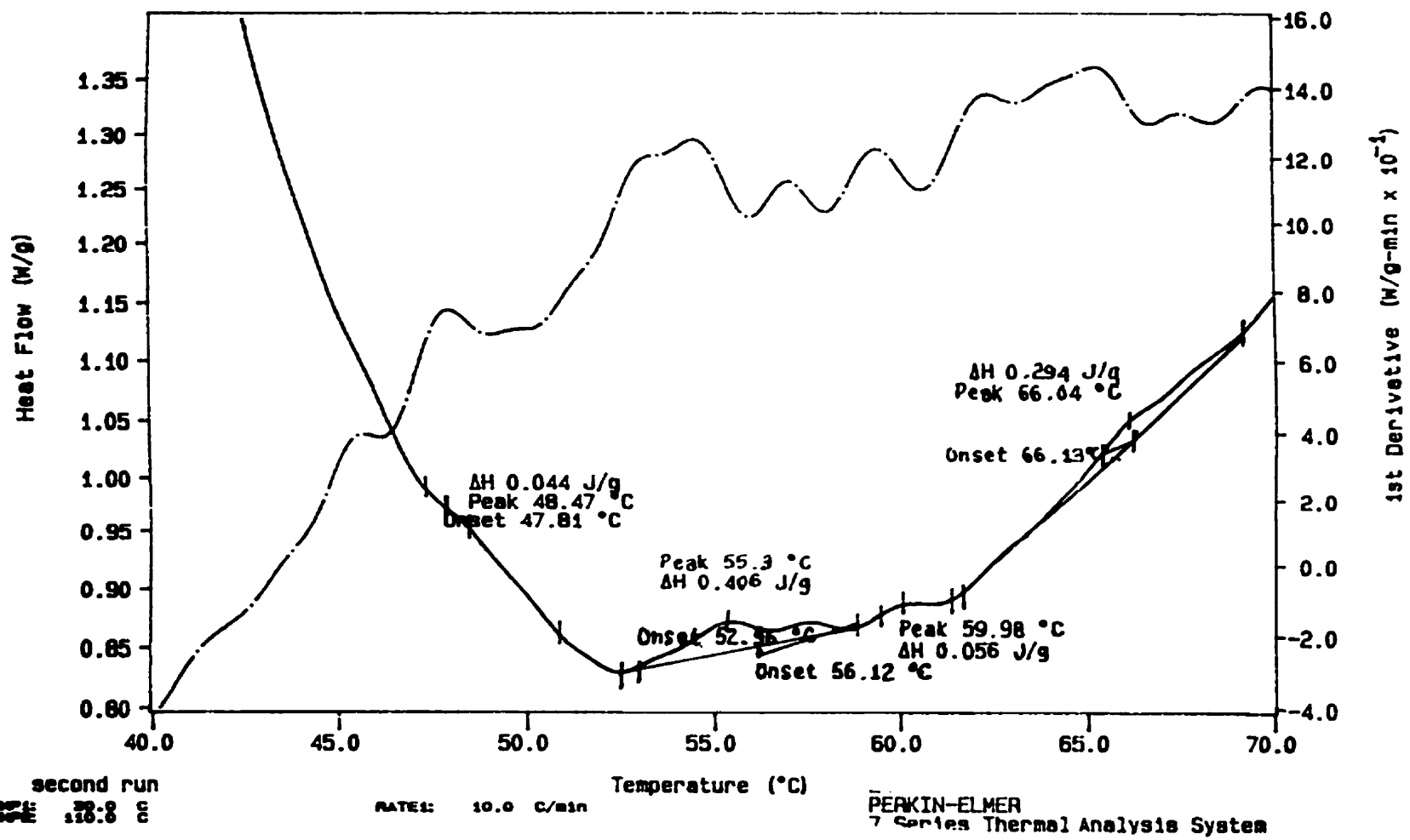
Treatment: 4.8kW/m²; 26% m.c.; Trial 2; 2wk after processing

File info: 10302a Thu Apr 22 12:36:03 1999
Sample Weight: 9.550 mg



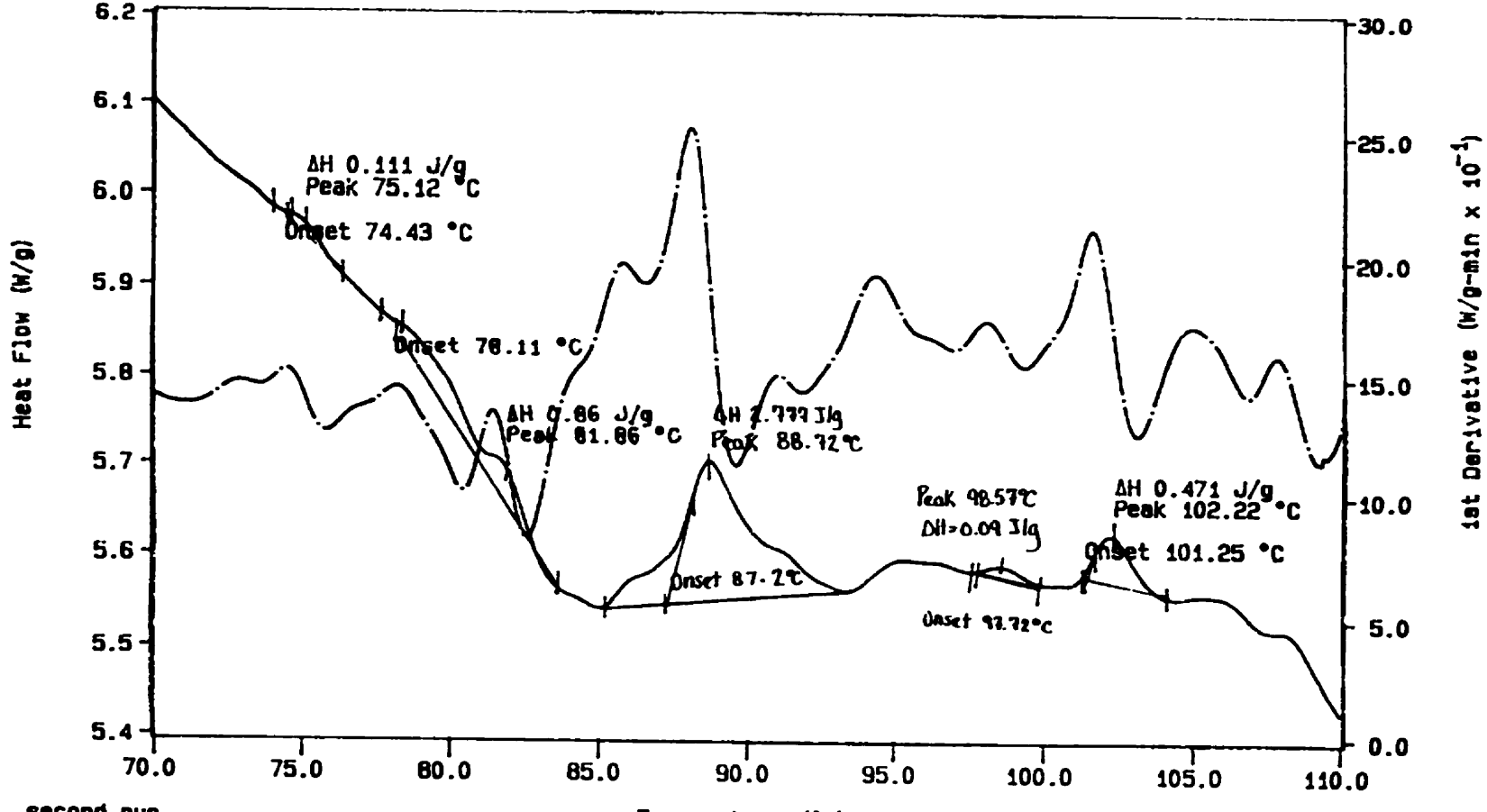
Treatment: 16.8 KW/m^2 ; 28% m.c. ; Trial 2

File info: 12283b Wed Apr 21 20:15:30 1999
Sample Weight: 7.360 mg



Treatment: 12.5KW/m²; 26% m.c.; Trial3

File info: 12283b Wed Apr 21 20:15:30 1999
Sample Weight: 7.360 mg



second run
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 10.0 C/min
TEMP2: 110.0 C

Treatment: 12.3 kW/m²; 26% m.c.; Trial 3

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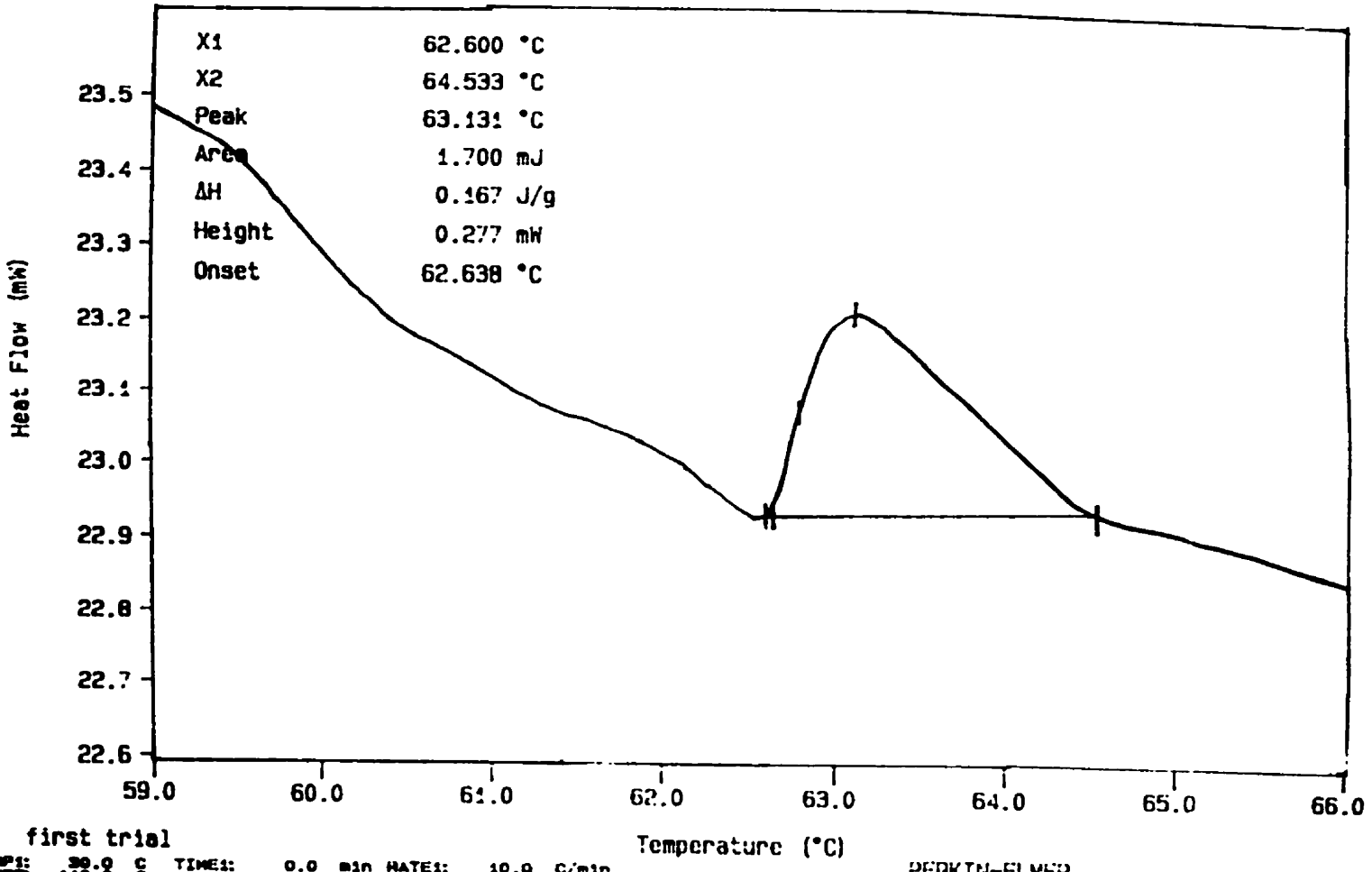
File info: 12302 Wed Apr 21 15:08:16 1999
Sample Weight: 10.180 mg

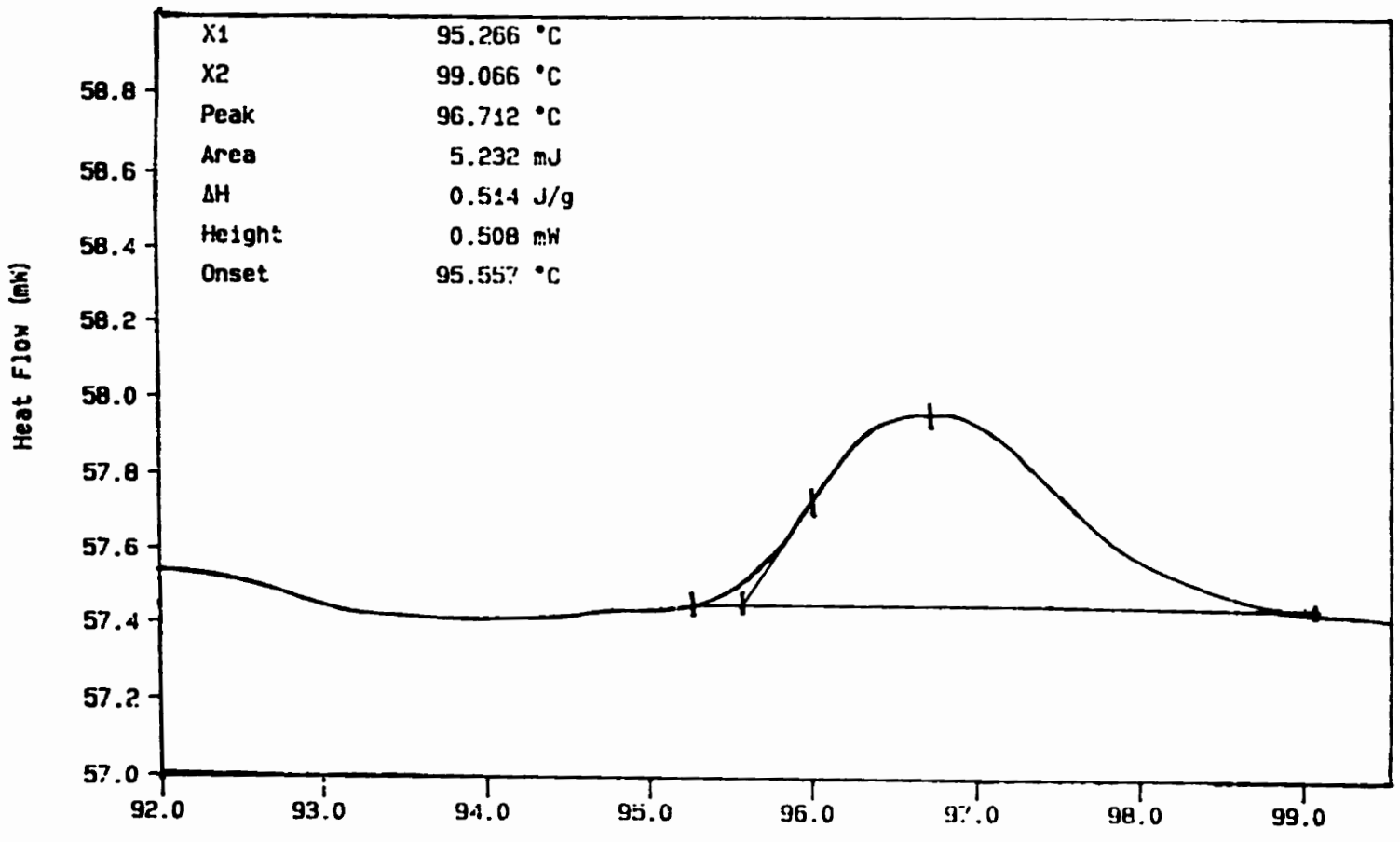


first trial
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 10.0 C/min
TEMP2: 110.0 C

Treatment: 12.3 kW/m^2 ; 28% m.c. ; Trial 2

PERKIN-ELMER
7 Series Thermal Analysis System

File info: 12302 Wed Apr 21 15:08:16 1999
Sample Weight: 10.180 mg



first trial
TEMP1: 30.0 C TIME1: 0.0 min RATE1: 10.0 C/min
TEMP2: 110.0 C
LW
PERKIN-ELMER
7 Series Thermal Analysis System

Treatment: 12.3 kW/m²; 28% m.c.; Trial 2

APPENDIX J DSC CURVE FOR MELTING WAXY MAIZE