

CHAPTER 13, SUBJECT 3

PROTOCOL FOR CARRYING OUT HEAT-PENETRATION STUDIES

Various methods and equipment may be employed in order to collect accurate heat-penetration data. The overall objective of these guidelines is to recommend procedures for carrying out heat penetration studies for establishing thermal processes necessary to produce commercially sterile foods packaged in hermetically sealed containers. **The following recommendations are to be considered voluntary guidelines.** While this does not preclude the application of other methods and equipment for collecting heat-penetration data, these guidelines have been developed by consensus of the Institute for Thermal Processing Specialists and should be given serious consideration for adoption as methodology by individuals performing heat-penetration studies.

1. NOMENCLATURE

t	Time
t _c	Retort come-up time is the time between the start of heating and the time when the retort reaches processing temperature (at times referred to as CUT)
t _p	Process time is the time from the end of the come-up period to the end of heating (at times referred to as the operator's process time)
T	Temperature
T _c	Container center or coldspot temperature (at times referred to as CT)
T _r	Retort temperature (at times referred to as RT)
T _w	Cooling water temperature (at times referred to as CW)

2. TERMINOLOGY

- 2.1 *Ballast Containers:* Containers may be required to fill the retort during heat-penetration studies to simulate production retort conditions. Type, shape and size of containers should be the same as used for the intended process. Material used for filling containers could be the test product, or any suitable material having heating characteristics similar to that of the test product, or in some circumstances, water.
- 2.2 *Cooling Time:* Time required following the introduction of the cooling medium to decrease the internal temperature of the product to a specified value, commonly 35 to 45+ °C (95 to 110+ °F).

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- 2.3 *Critical Factors:* Physical and chemical factors that can influence the thermal response of a product to a thermal process, the variation of which may influence the scheduled process, including: container, product, retort and processing conditions.
- 2.4 *Fill, Drain and Net Weights:* Fill weight is the weight of solids prior to processing; drain weight, the weight of solids after processing; and net weight, the weight of all product in a container.
- 2.5 *Heat-Penetration Curve:* Plot of the logarithmic difference between either retort temperature and product temperature (heating curve) or product temperature and cooling medium temperature (cooling curve) versus time.
- 2.6 *Mercury-in-Glass Thermometer (MIG):* Generally used as the retort reference temperature device and regulated for that application by government agencies in some countries. Other temperature-measuring devices may be calibrated against a MIG retort thermometer which has been calibrated against a traceable temperature standard.
- 2.7 *Resistance-Temperature Detector (RTD):* Thermometry system based on the positive change in resistance of a metal-sensing element (commonly platinum) with increasing temperature.
- 2.8 *Temperature-Measuring Device (TMD):* Device used for measuring temperature, including: thermometers, thermocouples, RTDs and thermistors.
- 2.9 *Thermistor:* TMD manufactured from semiconductor materials which exhibits large changes in resistance proportional to small changes in temperature. Thermistors are more sensitive to temperature changes than thermocouples or RTDs and are capable of detecting relatively small changes in temperature.
- 2.10 *Thermocouple:* TMD composed of two dissimilar metals which are joined together to form two junctions. When one junction is kept at an elevated temperature as compared to the other, a small thermoelectric voltage or electromotive force (emf) is generated which is proportional to the difference in temperature between the two junctions.

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3. DESIGN OF A HEAT-PENETRATION STUDY

The purpose of a heat-penetration study is to determine the heating and cooling behaviour of a product/package combination in a specific retort system for the establishment of safe thermal processes and evaluating process deviations. The study must be designed to adequately and accurately examine all critical factors associated with the product, package and process which affect heating rates. Numbers of containers per test run, and number of test runs to account for statistical variability are important and discussed in sections 5.11 and 5.12. Before commencing a heat-penetration study, an evaluation of retort temperature and heat transfer uniformity, at times referred to as a heat or temperature distribution study (IFTPS, 1992), should have been completed. A goal in conducting these studies is to identify the worst-case temperature response expected to occur in commercial production as influenced by the product, package and process.

4. FACTORS AFFECTING HEATING BEHAVIOUR

Several product, process, package and measurement-related factors can contribute to variations in the time-temperature data gathered during a heat-penetration test. Establishment of a process requires expert judgement and sound experimental data for determining which factors are critical and the effect of changing those factors both within and beyond established critical limits. The list of items addressed in this section is extensive, but should not be assumed to cover all possible factors. Quantitative data on variability should be recorded where appropriate and all pertinent data should be documented to better understand and account for possible variations in heat-penetration behaviour.

4.1 *Product:*

4.1.1 Product formulation and weight variation of ingredients should be consistent with worst-case production values. Changes in formulation may necessitate a new heat-penetration study.

4.1.2 Fill weight used for heat-penetration studies should not be less than the maximum declared on the process

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schedule. Excess product may be expressed as percent overflow.

- 4.1.3 Solids content should be measured for nonhomogeneous products both before and after processing. Solids content deposited in a sieve should be weighed and expressed as a percentage of total weight. Note: Addition of compressed or dehydrated ingredients may result in increased drained weight.
- 4.1.4 Consistency or viscosity of semi-liquid or liquid components should be measured before and after processing. Flow behaviour will change with type and concentration of thickening agent (starch, gums, etc.), temperature and shear rate. Changes may be reversible or irreversible which may be important when reprocessing product.
- 4.1.5 Size, shape and weight of solid components should be measured before and after processing.
- 4.1.6 Integrity and size of solid component clusters may change during processing and affect temperature sensor placement in the product and coldspot location.
- 4.1.7 Methods of product preparation prior to filling should simulate commercial practice. For example, blanching may cause swelling, matting or shrinkage which could influence heat-penetration characteristics.
- 4.1.8 Product matting or clumping may change heat-penetration characteristics and influence coldspot location. Also, caution should be exercised with sliced products which may stack together during processing.
- 4.1.9 Rehydration of dried components, either before or during processing, is a critical factor which may influence heat-penetration behaviour, as well as process efficacy with respect to spore inactivation. Details of rehydration procedures should be recorded during the heat-penetration study.
- 4.1.10 Product may heat by convection, conduction or mixed convection/conduction depending on its physical properties. Some foods exhibit complex (broken) heating behaviour. Product may initially heat by convection, then due to a physical change in the product, change to

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conduction-heating behaviour. For example, for products such as soups which contain starch, the change in heating behaviour may be due to starch gelatinization at a particular temperature. Small variations in product formulation or ingredients may cause the transition from convection to conduction heating to occur at a different temperature and related time. Special care should be taken to identify and control specific product and process variables related to the heating rates of these products.

- 4.1.11 Additional product characteristics such as salt content, water activity, pH, specific gravity, concentration of preservatives, and methods of acidification may influence heat transfer or microbiological resistance and should be recorded.
- 4.2 *Container:*
 - 4.2.1 Manufacturer and brand name information should be recorded in case information related to filling, sealing or processing is required.
 - 4.2.2 Container type (metal cans, glass jars, retort pouches, semi-rigid containers), size and dimensions should be recorded.
 - 4.2.3 Nesting of low profile containers can influence heating behaviour. Heat-penetration studies on jumble-loaded retorts (no racks or dividers) should include tests conducted on stacks of nested cans as well as single cans.
 - 4.2.4 Container vacuum and headspace should be recorded for rigid containers. For flexible and semi-rigid containers the volume of residual gases in the container should be determined. Entrapped gases may create an insulating layer in the container causing a shift in the coldspot location and a decrease in the heating rate. Controlled overpressures during processing have been found to reduce these effects.
 - 4.2.5 Maximum thickness of flexible packages (pouches) has a direct relationship to the coldspot temperature history with thicker packages heating more slowly. Heat-penetration studies should be carried out at the maximum specified package thickness.

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- 4.2.6 Container orientation (vertical or horizontal) within the retort may be a critical factor for some product/package combinations and should be studied where appropriate. Changes in container orientation may also influence vent schedules and come-up time.
- 4.2.7 Postprocessing examination of test containers for abnormalities should be conducted with special emphasis on the slowest and fastest heating containers. It is strongly recommended that flexible packages be carefully examined following processing to identify the thermocouple junction location. If the intended sensing location has shifted, it is likely that heat-penetration data collected are not reliable.
- 4.3 Method of Fill:
- 4.3.1 Fill temperature of the product should be controlled. It will affect the initial temperature which may influence some heat-penetration parameters (lag factor, retort come-up period). This may constitute a critical control point for a process, particularly for products which exhibit broken heating behaviour.
- 4.3.2 Fill and net weights may influence heating rates both in still and rotary cooks. Information on variability may be found in statistical process control and product quality control records.
- 4.3.3 In most cases, controlling headspace by determining net weight is not sufficient due to possible variations in the specific gravity of the food product. Care should be taken to avoid incorporation of air which would affect the headspace vacuum. In rotary processes, container headspace is a critical control point since the headspace bubble helps mix the product during agitation.
- 4.4 *Closing or Sealing:* Closing or sealing equipment should provide a strong, hermetic seal which is maintained during the thermal process. Vacuum in cans and jars for most canned foods is recommended to be between 35-70 kPa (10-20 in-Hg) measured at room temperature. Vacuum is affected by variables such as: headspace, product temperature, entrapped air, and vacuum efficiency of the closing equipment. Some products such as vegetables vacuum-packed in cans may have a minimum vacuum as a critical control point. For others packed in flexible or

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semi-rigid containers, vacuum setting will influence the residual air content in the package, also constituting a critical control point.

- 4.5 *Retort System:* The type of retort system used may have a significant influence on the heating rates of products processed in the retort. Results from a heat-penetration test should be reported with reference to the retort type and conditions existing at the time of testing.
- 4.5.1 Retort come-up time should be as short as possible, consistent with obtaining satisfactory temperature distribution. Laboratory size retorts may be used for development work on heat-penetration behaviour. Results will be conservative when the smaller retorts have shorter come-up times and cool more quickly than production retorts. After development, the thermal process should, if physically possible, be verified in an appropriate production retort.
- 4.5.2 Racking systems may be used to separate layers of cans or jars, constrain the expansion of semi-rigid and flexible containers, provide support and circulation channels for thin profile containers, and ensure maximum pouch thickness is not exceeded. Care should be taken to understand the influence of a specific rack design on retort performance and heat transfer to containers.
- 4.5.3 Still batch-retort systems vary in operation based on: type of heating medium (steam, steam/air, water immersion, water spray); orientation of the retort (vertical, horizontal); method of heating medium agitation (fans, pumps, air injection); and other factors which may influence the heating behaviour.
- 4.5.4 Rotational batch retort systems (axial, end-over-end) are designed to rotate (or oscillate) entire baskets of product during processing. Container agitation may provide faster rates of heat penetration to the container coldspot as compared to still cooks. However, while this is true for some containers, it may not be so for all containers within a load and caution must be exercised to identify the slowest heating containers. This may entail a detailed can position study. It is recommended that during initial testing, data be collected at small time increments (15 s) particularly for viscous fluids where the coldspot may move in relationship to a fixed

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thermocouple during rotation, producing erroneous results. Slip-ring connectors should be cleaned and thermocouple calibration verified at regular intervals. Critical factors in these retorts include: headspace, product consistency, solids to liquid ratio, initial temperature, container size, rotational speed and radius of rotation.

- 4.5.5 Continuous retort systems may move containers through the processing vessel along a spiral track located at the outside circumference of a horizontal retort shell or be carried through a hydrostatic retort in chain driven flights. Regardless of the configuration, it becomes difficult or impossible to use thermocouples to collect heat-penetration data in these systems. Data may be obtained using self-contained temperature measurement and data storage modules in the commercial vessel or by using process simulators.

5. TEMPERATURE MEASUREMENT AND DATA ACQUISITION

- 5.1 *Data Acquisition System:* Accuracy and precision of the data acquisition system (datalogger) used for heat-penetration studies will affect temperature readings. Dataloggers are typically comprised of a multi-channel temperature-measuring and digital-data-output system. Calibration of a data-acquisition system should include verification of the data-acquisition rate, since errors in the time base would result in erroneous data.
- 5.2 *Type of Thermocouple:* The most common TMDs used in thermal processing are duplex Type T (copper/constantan) thermocouples with Teflon insulation. Common configurations are flexible wires (20-, 22- or 24-gauge) and rigid needle types. Details on thermocouples and connecting units are available in Bee and Park (1978) and Pflug (1975).
- 5.3 *Type of Connectors and Associated Errors:* Connectors used in a thermocouple circuit are fittings attached to a thermocouple within which electrical connections are made. Several types of connectors are available for specific applications and thermocouple type. Caution must be exercised to avoid certain sources of error which may be associated with the use of connectors and extension wires. These include: disparity in thermal emf between

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thermocouples, connectors and extension wires; temperature differences between two wire junctions; and reversed polarity at the thermocouple-extension wire junction. Thermocouple connectors should be cleaned frequently with metal cleaner to assure good electrical contact and prevent errors in thermocouple readings. Similar concerns should be addressed when using RTDs and thermistors.

- 5.4 *Thermocouple Calibration:* Thermocouples should be calibrated against a traceable calibration standard (thermometer, RTD, thermistor). Inaccuracies in temperature measurements may result in errors in process evaluation; hence, frequent calibration is essential to provide reliable data. Factors affecting calibration include: worn or dirty slip-rings; improper junctions; metal oxidation; multiple connectors on one lead, and inadequate datalogger cold junction compensation. As a consequence, thermocouples should be calibrated in place as part of the complete data-acquisition system. Some precautions when using thermocouple-based data-acquisition systems include: minimizing multiple connections on the same wire; cleaning all connections; grounding the thermocouples and recording device; slitting thermocouple outer insulation outside the retort to prevent flooding of datalogger or data recording device (see NFPA, 1985, or ASTM, 1988 for illustrations); and using properly insulated thermocouple wires.
- 5.5 *Positioning of Thermocouple in the Container:* The method of inserting a thermocouple into a container should result in an airtight, watertight seal which should be verified after testing. Thermocouple sensing junctions should be positioned in the slowest heating component of the food product and situated in the slowest heating zone within the container. During insertion of the thermocouple, caution must be taken to avoid physical changes to the product. Also, the method employed for mounting the thermocouple into the container should not affect the container geometry which could influence heat-penetration characteristics. Flexible or rigid thermocouples may be inserted into rigid, flexible and semi-rigid containers using compression fittings or packing glands. For flexible containers, NFPA (1985) provides illustrations of thermocouple positioning into a solid particulate and several thermocouple positioning devices to ensure the thermocouple remains in a fixed

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position within the container. The most appropriate device for a particular application will depend upon the product, racking system, container type and sealing equipment. Leakage may be detected by weighing the container before and after processing to determine changes in gross weight. If there is leakage caused by improperly mounted thermocouples, data collected for that container should be discarded. Note: Ecklund (1956) reported correction factors for heat-penetration data to compensate for errors associated with the use of non-projecting, stainless steel receptacles. While not reported in the literature, this may also be a concern with other fittings.

- 5.6 *Type and Placement of Containers:* The type and size of container used in the heat-penetration study should be the same as that used for the commercial product. The racking and loading of rigid (cans), semi-rigid (trays and cups) and flexible (pouches) containers should simulate commercial practice. Test containers should be placed at the slowest heating location in the retort, as determined by temperature and heat transfer distribution studies.
- 5.7 *Temperature of the Heating Medium:* TMDs should be positioned so as to prevent direct contact with racks or containers and identified according to their specific location in the retort. A minimum of two thermocouples is recommended for retort temperature measurement: one situated close to the sensing bulb of the retort MIG thermometer, the other located near the test containers. In addition, at least one thermocouple should be placed near the sensor for the temperature controller when that location is remote from the location of the MIG thermometer bulb.
- 5.8 *Retort Pressure:* Overpressure conditions during processing will influence package expansion by constraining the expansion of headspace gases. This may be beneficial by improving heat transfer to food in flexible and semi-rigid containers or detrimental by restricting the size of the headspace bubble in rotary processes. For steam/air retorts, overpressure conditions are also related to the steam content of the heating medium at a particular processing temperature which may influence heat transfer conditions within the retort. In addition, cooling without overpressure may result in

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depressurization within a container upon collapse of steam at the end of a process, leading to accelerated decreases in temperature for fluid foods.

- 5.9 *Coldspot Determination:* The location of the slowest heating or coldspot in a container is critical to establishing a process. For a conduction heating product in a cylindrical can with minimal headspace, the geometric center of the can is considered to be the slowest heating spot. Generally, if a larger headspace is included, the coldspot may shift closer to the top of the can due to the insulating effect of the headspace which may be significant if the height-to-diameter ratio of the can is small. The coldspot location in vertically oriented cylindrical cans containing products which heat by natural convection may be near the bottom of the container. Products which exhibit broken heating behaviour may have a coldspot which migrates during heat processing as the physical properties of the product change. The use of containers with different geometries or constructed from different materials may have differing effects on coldspot locations. A coldspot-location study should be completed to determine the slowest heating location for a specific product/package/process combination. Usually, the coldspot location will be determined from a series of heat-penetration tests employing several containers with thermocouples inserted at different locations. Alternatively, more than one thermocouple per container may be used; however, multiple thermocouples may influence heating behaviour, especially for products in smaller containers. In all cases, care should be taken to determine the "worst case" anticipated during production. Careful judgement, based on a number of preliminary experiments, must be exercised to ensure the coldspot location has been identified.
- 5.10 *Initial Product Temperature:* Measurement of initial product temperature should be taken immediately prior to testing.
- 5.11 *Number of Containers per Test Run:* A heat-penetration test should evaluate at least 10 working thermocouples from each test run (NFPA, 1985). If the retort cannot accommodate this quantity, the number of replicate test runs should be increased.

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5.12 *Number of Test Runs:* Replication of heat-penetration test runs is important in order to obtain results which account for run-to-run, product, container and process variability. After initial coldspot-determination tests are completed and all critical factors have been determined, at least two full replications of each test are recommended. Should results from these tests show variation, a minimum of a third test is recommended. Variation in the results is expected and quite common, especially for products which are non-homogeneous or exhibit complex heating behaviour. Variability is generally evaluated based on plots of the heating and cooling curves and/or lethality calculations and should be considered when identifying or predicting the slowest heat behaviour of a process

6.0 SUMMARY OF DOCUMENTATION

The following provides a summary of details which may be incorporated in a checklist and documented in their entirety or partially as deemed appropriate for a specific study. Other factors not listed in this section may also be relevant.

6.1 *Pre-test Documentation:*

6.1.1 Product Characteristics

- 6.1.1.1 Product name, form or style, and packing medium
- 6.1.1.2 Product formulation and weight distribution of components
- 6.1.1.3 Net weight and volume
- 6.1.1.4 Consistency or viscosity of the liquid component
- 6.1.1.5 Size, shape and weight of solid components
- 6.1.1.6 Size of solid component clusters
- 6.1.1.7 pH of solid and liquid components
- 6.1.1.8 Methods of preparation prior to filling (ingredient mixing methods, special equipment)
- 6.1.1.9 Matting tendency
- 6.1.1.10 Rehydration of components
- 6.1.1.11 Acidification procedures
- 6.1.1.12 Other characteristics (% solids, density, etc.)

6.1.2 Container Description

- 6.1.2.1 Container material (brand name and manufacturer)
- 6.1.2.2 Type, size and inside dimensions
- 6.1.2.3 Container test-identification code
- 6.1.2.4 Maximum thickness (flexible container)
- 6.1.2.5 Gross weight of container

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- 6.1.2.6 Container nesting characteristics
- 6.1.2.7 Slowest heating or coldspot location in container
- 6.1.3 Data-Acquisition Equipment and Methodology
 - 6.1.3.1 Identification of datalogging system
 - 6.1.3.2 Thermocouple and connector plugs maintenance
 - 6.1.3.3 Thermocouples and connectors numbered
 - 6.1.3.4 Electrical ground checked
 - 6.1.3.5 Thermocouples placed in heating medium and readings compared with a reference TMD
 - 6.1.3.6 Type, length, manufacturer and identification code of thermocouples and connectors
 - 6.1.3.7 Thermocouple location in container
 - 6.1.3.8 Positioning technique for thermocouple
 - 6.1.3.9 Calibration data for each thermocouple
- 6.1.4 Fill Method
 - 6.1.4.1 Fill temperature of product
 - 6.1.4.2 Fill weight of product
 - 6.1.4.3 Headspace
 - 6.1.4.4 Filling method (comparison to commercial process)
- 6.1.5 Sealing Operations
 - 6.1.5.1 Type of sealing equipment
 - 6.1.5.2 Time, temperature, pressure and vacuum settings (if applicable)
 - 6.1.5.3 Gas evacuation method
 - 6.1.5.4 Can vacuum
 - 6.1.5.5 Volume of residual gases in flexible containers
- 6.1.6 Retort System
 - 6.1.6.1 Retort system: still or rotary (end-over-end, axial, oscillatory)
 - 6.1.6.2 Reel diameter (number of container positions) and rotational speed
 - 6.1.6.3 Can-position study data for batch rotary retorts
 - 6.1.6.4 Heating medium (steam, steam/air, water immersion, water spray) and flow rate
 - 6.1.6.5 Circulation method for water or overpressure media
 - 6.1.6.6 Temperature distribution records
 - 6.1.6.7 Retort venting schedule
 - 6.1.6.8 Retort identification number
- 6.1.7 Loading of Retort
 - 6.1.7.1 Loading or racking system details
 - 6.1.7.2 Location of test containers in retort (slowest heating zone)
 - 6.1.7.3 Container orientation
 - 6.1.7.4 Location of thermocouples for retort temperature measurement
 - 6.1.7.5 Use of ballast containers to ensure fully loaded retort (some retort systems)

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- 6.1.7.6 Selected time interval for data-logging system
- 6.1.8 Additional Information
 - 6.1.8.1 Date
 - 6.1.8.2 Test identification
 - 6.1.8.3 Processor and location
 - 6.1.8.4 Individual(s) performing heat-penetration test
- 6.2 *Test-Phase Documentation:*
 - 6.2.1 Test run identification
 - 6.2.2 Initial temperature of product at the start of heating
 - 6.2.3 Time heating starts
 - 6.2.4 Time vent closed and temperature, if applicable
 - 6.2.5 Temperature indicated on MTG thermometer
 - 6.2.6 Time retort reaches set point temperature (tc)
 - 6.2.7 Pressure from a calibrated pressure gauge or transducer
 - 6.2.8 Time process begins
 - 6.2.9 Time cooling begins (pressure cooling, if applicable)
 - 6.2.10 Time cooling ends
 - 6.2.11 Rotation speed (if applicable)
 - 6.2.12 Cooling water temperature
 - 6.2.13 Any process irregularities or inconsistencies
- 6.3 *Post-Test documentation:*
 - 6.3.1 Container net and gross weight check for leakage
 - 6.3.2 Thickness of container
 - 6.3.3 Location of the thermocouple and whether or not it is impaled in a food particle
 - 6.3.4 Measurement of container vacuum (rigid metal and glass) or residual air content (flexible and semi-rigid containers)
 - 6.3.5 Post-processing product characteristics: syrup strength, appearance, viscosity, headspace, drained weight, pH, consistency, shrinkage, matting, clumping
 - 6.3.6 Container location and orientation (jumble pack)

7. LITERATURE CITED

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Institute For Thermal Processing Specialists
P.O. Box 2764 · Fairfax, VA 22031-0764
Telephone: (703) 591-1108 · Fax (703) 591-5903