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Tin-Filled Multi-Entrance Fixed Point

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Abstract. In this paper, the advantages, design possibilities, performance of the Multi-Entrance Fixed Point (MEFP) concept are described. The purpose of MEFP is to replace equalizing blocks in any temperature-controlled zone with phase transition of matter during melting or freezing. The phase transition in such a calibration is not intended to be the temperature standard itself, but only provides for spatial thermal homogeneity and temporal stability during comparison calibration. Knowing that some of the main uncertainty components in the uncertainty budget of the comparison calibrated thermometer are the radial and axial gradients in the isothermal zone and knowing that comparisons comprise by far the majority of all calibrations, the improvement of speed and quality of calibration is of significant importance. The tin-filled MEFP with multiple entrance tubes, designed in the Laboratory for Process Measurements allows for simultaneous calibration of several thermometers against a standard thermometer. MEFP prototypes filled with other materials and with other design characteristics are pending production. This concept allows the use of modest purity materials to fill the cell, since a standard thermometer is used as the calibration reference. During this investigation, thermometers were calibrated against a standard in the same furnace, using both the standard equalizing block and the MEFP. The measurement results indicate that the use of MEFP decreases the uncertainty contributions from temperature gradients and the stability of the bath by an order of magnitude, compared to the standard equalizing block.

Keywords: comparison calibration, equalizing block, Multi-Entrance Fixed Point.

INTRODUCTION

Dissemination of the International Temperature Scale (ITS90) to the lower-grade Standards and thermometers in Croatia [1] is, as in other countries, accomplished by the comparison calibrations which comprise by far the majority of all calibrations. The hard-won and costly improvements in the temperature fixed points realizations, measured in microkelvins, are "diluted" down the traceability chain in much larger uncertainties present at comparison calibrations. Therefore improvement of speed and quality of comparison calibrations is of significant importance. One of the main uncertainty components in the uncertainty budget of the comparison calibrated thermometers are the radial and axial gradients in the isothermal zone. When using a high-precision bridge for calibration of several thermometers, stability of the bath/furnace takes an important role as bridge balance time of approx. 20 s between readings can cause a significant time shift between reading standard thermometer and thermometers under test. Low uncertainty comparison calibrations are generally performed either by bath/furnace with advanced temperature controlling system or by a furnace with heat pipe, with both solutions being expensive. In order to improve comparison calibration capability using an available set of equipment and minimal additional investment, a principle of phase transition was adopted for comparison calibration under the name Multi-Entrance Fixed Point (MEFP). Unlike in ITS-90 temperature fixed points, phase transition in such calibrations was not intended to be a temperature standard by itself, but only provides for thermal homogeneity, both spatial and in time. This concept allows for use of modest purity materials for filling the cell, since standard thermometer is used as the calibration reference. For the same reason, there is no need to regulate or measure absolute pressure during phase transition. Bath or furnace with lower grade controller performance, spatial gradients and stability can be used as the phase transition, i.e. solid-liquid interface, will provide ample temperature stability and uniformity.

The first MEFP cell intended for comparison calibration was built and investigated by Laboratory for process measurement (LPM). It was designed in a form of four thermometer entrance tubes, immersed into a cylindrical, stainless steel vessel filled with mercury [2]. Cell was completely sealed due to health risk involved in mercury handling. During investigation, thermometers were calibrated against a standard in the same bath, using both the standard equalizing block and the MEFP. The measurement results clearly indicate that use of the MEFP decreases the uncertainty contributions from temperature gradients and the stability of the bath by an order of magnitude, compared to the equalizing block.

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Temperature stability of the bath was decreased from 15 mK to 0.5 mK over a time period of 120 min and thermal gradients from 6 mK to less than 0.5 mK in the same time interval.

As tests with mercury-filled MEFP yielded good results, LPM continued with development of the concept. The cells intended for use at elevated temperatures were of interest as comparison calibration furnaces in general have greater spatial and temporal temperature inhomogeneity than baths. It was decided that the next step would be the design, construction, filling and investigation of the tin-filled MEFP Although commercial liquid baths with temperatures sufficiently high for melting of tin are available, it was decided to use the furnace for the tin-filled MEFP investigations. Usefulness of the mercury-filled MEFP for improving thermal stability and uniformity of a liquid bath was already confirmed by previous research. The goal this time was to investigate what results the MEFP would produce in a harsher thermal environment, with lower temperature stability and uniformity. The MEFP cells of the same design will also be used for higher temperatures, where temperature gradients are main uncertainty components.

The main reason for the changes in design as opposed to the mercury-filled MEFP [2] was the risk of tin leakage in case of formation of cracks due to aggressive corrosion caused by molten tin at elevated temperatures. For this reason four reentrant tubes, used in the mercury cell, were replaced by more robust equalizing block. Besides robustness, equalizing block was easier to obtain than exceptionally thick walled closed metal tubes that would be required in order for the tin-filled MEFP to have acceptable life-span. Although there was also a possibility to use ceramic or graphite tubes for thermometer accommodation, it was abandoned because risk of crushing as a consequence of different thermal expansions of ceramic and tin. As the equalizing block used in the MEFP cell has a hole drilled through its center, it is lighter, has lower heat capacity and responds faster to temperature changes than molten or cured tin, which would be trapped between reentrant tubes, in case they were used, since phase transition always advances from outer walls towards MEFP center.

DESIGN AND FABRICATION OF THE TIN-FILLED MEFP CELL

The tin-filled MEFP cell was designed in a form of a within-the-cell equalizing block immersed into the tin-filled cylindrical vessel, as shown in Figure 1. Gray cast iron was used for manufacturing the cell. All cast irons contain more than 2% C. About 2% is the maximum C content at which iron can solidify as a single phase alloy with the entire C in solution in austenite. Thus, the cast irons by definition solidify as heterogeneous alloys and always have more than one constituent in their microstructure. In addition to C, cast irons also must contain appreciable silicon (Si), usually from 1–3%, and thus they are actually iron-carbon-silicon alloys. The high C content and the Si in cast irons make them excellent casting alloys. Although graphite was first taken into consideration for the cell material because of its inert properties, it was later decided to use gray cast iron instead. Some of the reasons were difficulties while attempting to buy and process relatively pure graphite in short period of time. Other reason was higher mechanical strength of gray cast iron cell (tensile strength of about 250 MPa, compression strengths 1000 MPa) and greater weight (7.36 kg/m^3) of the gray-cast iron equalizing block. This is important for preventing equalizing block to lift up due to effect of buoyancy, when immersed into a molten tin (6.99 kg/m^3). On the other hand, using metal container for containment of tin involves risk of the corrosion effects, especially at higher temperatures. After some period of use, corrosion could lead to tin leakage through the container or equalizing block towards a furnace or thermometers, possibly causing their permanent damage. For this reason, the MEFP cell was designed in such a way that there always exists at least 5 mm thick layer of gray cast iron between tin and thermometers and between tin and furnace. All edges which are in contact with tin are rounded to at least 4 mm diameter.

The tin container (Figure 1) is designed as a simple cylindrical vessel with outer diameter of 90 mm, 180 mm high and 5 mm thick wall. Equalizing block and tin vessel top cover are made as one part, preserving this way simplicity of design and to keep manufacturing costs low. The top cover is constructed as two concentric cylindrical plates, each 5 mm thick, which are meant to protect tin from pollution and to ensure optimal position of equalizing block inside the tin vessel. Upper plate lies on the top of the tin vessel and covers it completely, while a lower plate enters inside, close to vessel's inner walls, assuring this way central position of the equalizing block inside the molten tin. The bottom of the lower plate continues to form the conically shaped equalizing block. There are eight holes drilled through the cover plate of the MEFP, between the equalizing block and the plate edge. Four of them are intended for the installation of screws with hook, which serve for removing the equalizing block from the tin vessel, when the tin is molten. Other four holes are meant as prevention against pressure increase inside the cell during tin melting and eventual heating to the temperatures above its melting point. Those holes have 6 mm inner diameter. Equalizing block is provided with four borings used to accommodate any combination of standard and test thermometers.

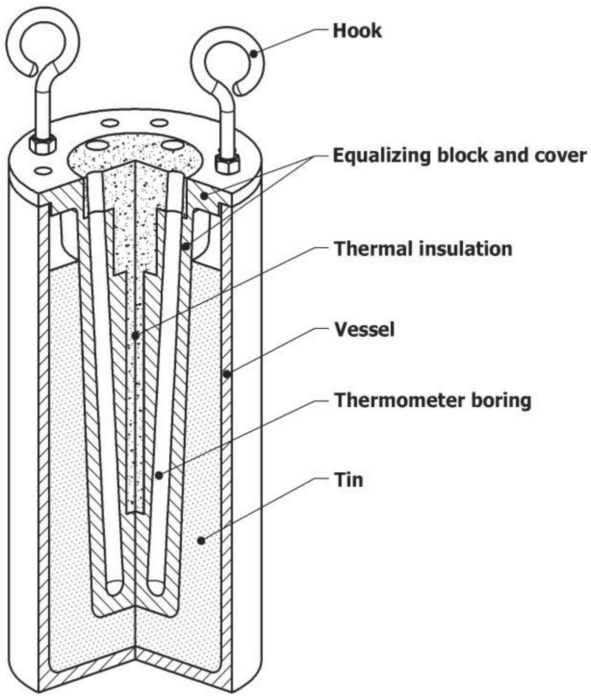

FIGURE 1. Schematic of the tin-filled Multi-Entrance Fixed Point.

All borings are of 8 mm inner diameter, 160 mm deep and all are angled 7 degrees from vertical axis, assuring this way more space for the thermometer's heads. Distance between thermometer's borings is 38 mm at the top and 20 mm at the bottom of the block. This design insures that the thermometer sensors, at the bottom of the borings, are surrounded by the largest amount of tin, preventing this way formation of a 'bridge' during the phase transition. In order to decrease the weight of the equalizing block, and its heat capacity accordingly, one more boring was drilled through the center of the block. Decrease of the thermal capacity of the block results in a decrease of its thermal response time, providing faster stabilization of thermometers under test. To prevent air circulation through the central hole and, in general, heat transfer between the cover of the equalizing block and the thermometers, the central hole was filled with thermal insulating material.

Prior to filling with tin, all surfaces of the MEFP cell and the equalizing block were cleaned with alcohol. As phase transition temperature inside the cell is to be measured with a calibrated SPRT, no special care was taken to remove all residual traces from the cell that might contaminate the tin and affect temperature transition. The filling of the cell was conducted by a local company, which also provided 99.95% pure tin for the purpose. Process of filling was conducted in the way that tin ingot was molten above the cell vessel, using an open flame burner. Although this method would induce additional contamination of tin, it was decided to adopt it, since the phase transition temperature will be monitored with calibrated SPRT. The cell was filled with approximately four kilograms of tin and equalizing block was immediately immersed into a molten tin. This amount of tin was sufficient to form a column 150 mm high around the equalizing block and at least 15 mm of tin between the bottom of the cell and the bottom of the equalizing block.

CONTROLLED TEMPERATURE ZONE AND MEASURING EQUIPMENT

Relatively simple vertical cylindrical tube furnace was built to accommodate the tin-filled MEFP forming a compact, portable calibration unit. Furnace was basically made of three electrical heating elements, which are wound around a 400 mm long ceramic tube with an inner diameter of 120 mm. Electric heaters are 115 mm long, covering together 350 mm long section of the tube. Each heater is of one kilowatt nominal power and has a separate electric connector, allowing this way the use of three separate temperature controllers. Depending on the purpose, all heaters can be easily connected together to the same controller, either in parallel or serial connection.

During the measurements, three temperature controllers were used, in combination with industrial thin-film platinum resistance thermometers. While two of the controllers were produced by one company and use a solid-state relay as power output element, third controller is made by different company and uses a triac for powering lower heater of the furnace. Temperature controlling thermometers are positioned between the MEFP cell and furnace tube wall at different heights from the furnace bottom. After some preliminary experiments and determination of furnace axial temperature gradients, the MEFP cell was placed at about 80 mm above the furnace floor, using 60 mm long metal pipe of 80 mm diameter as support. In order to decrease heat loss through the bottom wall of the cell, 5 mm wide cylindrical copper plate and 10 mm wide layer of thermally insulation wool were placed between support and the bottom wall of the cell. Copper plate has 100 mm diameter, and it is meant to collect heat from furnace walls and improve heating of the cell bottom.

The temperature measurements were performed using two metal sheathed 25.5 Ω SPRTs and two ordinary metal-sheathed Pt 100s. All thermometers had a stem diameter of 6 mm. The thermometers resistances were measured with an ASL F700B AC Resistance Bridge, which when used with a 100 Ω standard resistor, has a resolution of 1 mK for a 25.5 Ω SPRT and 0.3 mK for Pt 100. The bridge was connected to a 10-channel scanner, and all the data were collected through an IEEE-488 connection to a computer. The average balance time for the bridge was 20 s, allowing approximately 80 s for the full cycle with four thermometers. The standard resistor used was Wilkins-type 100 Ω and it was kept immersed in thermostated oil bath at temperature of 23 °C.

MEASUREMENT RESULTS

All measurements in the MEFP cell were performed using the same furnace, thermometers and resistance bridge. The data were collected in the same way to avoid biasing the results.

Four thermometers were placed in their respective holes. Heat-conveying means such as liquids or bushings were not used. Due to that fact, there was an air gap of about 2 mm between the thermometers and the MEFP borings, as a result of difference between MEFP diameters. For thermometers with a stem diameter diameter smaller than 6 mm, use of heat-conveying means is advisable. High-temperature insulation wool was used for thermal insulation of a thermometer stem protruding outside the cell. Approximately, 5 mm thick layer of the same type of insulation wool was also applied to the top cover of the MEFP cell, in order to decrease heat loss and axial temperature gradients inside the equalizing block, since the cell cover and equalizing block are made from one part, without thermal insulation in-between.

Although MEFP cell was intended for temperature measurements during melting of tin, first measurement was taken at temperature below tin melting point. This was done in order to get information about performance of the furnace and the cell, when using the cell as an ordinary equalizing block. Obtained data are to be compared against performance of the cell during phase transition.

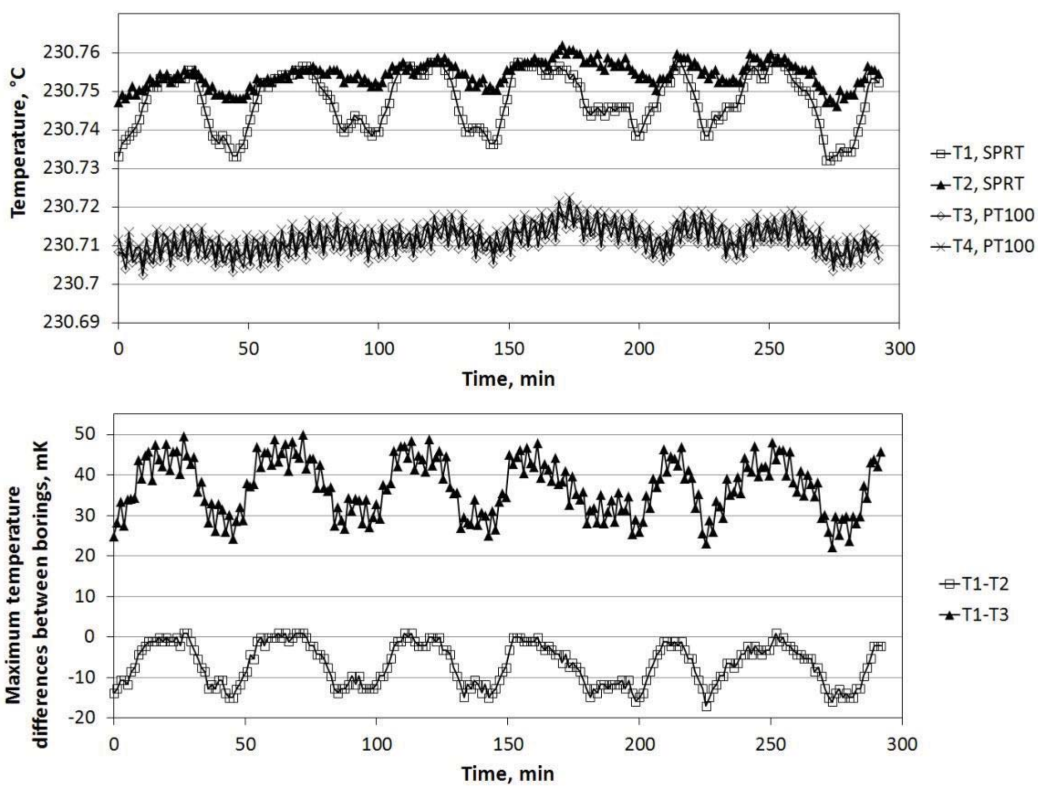

FIGURE 2. Stability and gradients in the MEFP when used as ordinary equalizing block, without phase transition.

After allowing a time for furnace to stabilize at temperature set-point of about 230.7 °C, temperature recording was started. In each measurement cycle, all four thermometers were read after 20 s of bridge balance time. In total, 220 measurements were taken within a period of approximately 5 hours.

Results of the temperature stability and gradients investigations determined in the described way above (when using the cell as an ordinary equalizing block, i.e. without phase transition) are presented in Figure 2. The readings of all the thermometers were oscillating within boundaries of 26 mK, due to instability of the furnace. The maximum differences between temperatures measured inside the thermometer borings, were in the range between 2 mK and 49 mK. Standard measurement uncertainty of the investigations of the temperature stability was estimated to 1.5 mK and standard uncertainty of the temperature differences between borings was estimated to 8 mK.

Thermal gradients outside the MEFP cell were roughly estimated to ±1 °C and thermal stability to 0.2 °C, based on results of separate investigation of vertical gradients inside the furnace.

After completion of the above described measurements at the temperature below melting point of tin, furnace was adjusted to temperature set-point of 232.3 °C (which is approximately 0.5 °C above tin melting point), without any other changes. The purpose of the furnace set point 0.5 °C higher than the melting point of tin was to achieve long duration of the melting plateau and to leave, in this way, enough time for MEFP cell to reach the temperature of the melting plateau. As MEFP cell has relatively high mass and high heat capacity accordingly, care should be taken to ensure it reaches melting temperature before phase transition comes to the end. This can be further minimized by setting furnace to the temperature slightly below the melting point first, then leaving MEFP cell to stabilize at this temperature and finally rising furnace temperature set point to desired value, above the melting point. No inner melt around thermometer borings was initiated, leaving only melting phase transition front to advance from the outer walls of the cell toward the equalizing block. Results of the MEFP cell testing are presented in Figure 3.

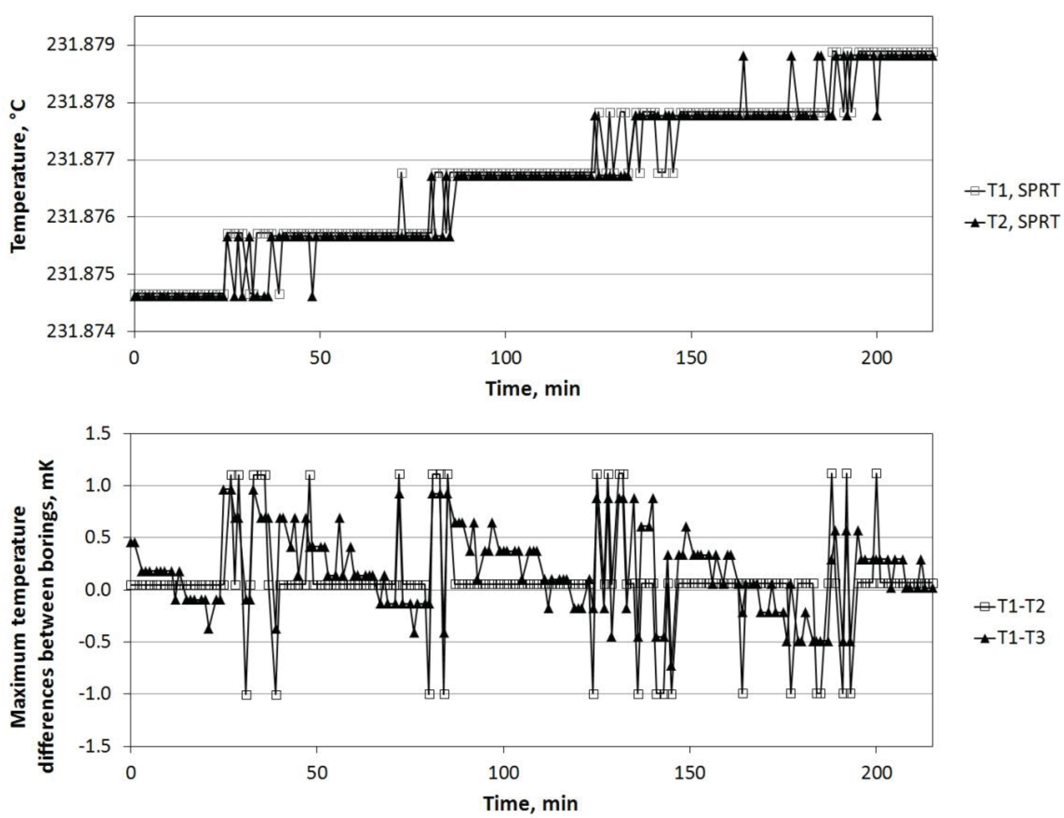

FIGURE 3. Stability and gradients in the MEFP during melting.

After a stabilization period, the thermometers in the MEFP cell showed stability of 5 mK over a period of 215 min and 1 mK for a duration of 75 min. Maximum temperature difference detected by SPRTs between two opposite MEFP borings was 2 mK. Although constant temperature increase of approximately 1 mK per hour can be observed on the presented chart, it is not significant, as reading of 10 measurements of all four thermometers, which is enough for comparison calibration, should not take more than 30 minutes. Standard measurement uncertainty of the stability determinations is estimated to 1.5 mK.

To limit measurement uncertainty and confirm the results of investigation of temperature differences between borings, additional test was performed in a same melting realization. Temperature difference was determined by exchanging thermometers between two opposite borings, according to the method described in the Euramet calibration guide "Calibration of Temperature Block Calibrators" [3]. Both thermometers used for the purpose were calibrated 25.5 Ω SPRTs. Temperature difference between borings is calculated based on the change of difference between readings of the two thermometers. By this method, most of the readings discrepancies should cancel out, decreasing this way overall uncertainty of the obtained result. Due to small drift of used equipment (SPRTs, resistance bridge and fixed resistor), over the few hours required for presented investigation, uncertainty components related to drifting of equipment could also be neglected. Performed test revealed temperature gradients of 1 mK and confirmed previously obtained results. Standard measurement uncertainty of the described determinations of the difference between borings was estimated to 2 mK.

All the uncertainties estimations stated included the:

- accuracy of the resistance bridge (together with bridge resolution),
- short term stability of the fixed resistor,
- stability of the bath where fixed resistor was kept,
- and stability of the SPRTs used.

Investigation of the cell behavior during the freezing of tin was not performed due to problems with supercooling, which was approximately 1 °C. The tin oxides layer, which rests on the top of the MEFP vessel, provides an excellent starting point for the nucleation, and formation of the bridge across a liquid-solid interface on the top of the cell accordingly.

CONCLUSION AND PERSPECTIVES

During the testing, the concept of the MEFP revealed both, the advantages and disadvantages. By using the MEFP cell instead of standard equalizing block, uncertainties due to thermal gradients and instabilities of the furnace at 230 °C were decreased by an order of magnitude. Other advantages are relatively small cost of both, the MEFP cell and the furnace, due to their relatively simple design. On the other hand, the disadvantages are that the phase transition in the MEFP cell, unlike the heat pipe, occurs only at single temperature, and unlike the fixed point, a calibrated standard is needed. The first issue can be solved by production of several MEFP cells, filled with different materials, in order to cover a certain temperature range. Low melting-point alloys used for prototyping in industry are available at low cost, with melting points temperatures ranging from 57 °C to 300 °C, depending on the composition. Only materials with single melting point are suitable for filling the MEFP cells, as for example in the low-range alloys based on gallium, bismuth (47 °C, 58 °C, 60 °C 70 °C, 79 °C, 95 °C, 100 °C, 109 °C, 124 °C, 135 °C, 138 °C), indium (47 °C, 58 °C, 79 °C, 93 °C, 118 °C, 123 °C, 143 °C, 147 °C, 157 °C), tin (145 °C, 162 °C, 221 °C, 183 °C), and lead (304°C, 309 °C, 327 °C) [4-6]. For higher temperatures, eutectic alloys based on zinc, gold, and aluminum should be investigated.

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