INL/EXT-20-59983
Revision 0

CEM MARS 6 Microwave Incident

September 2020

Changing the World's Energy Future

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Idaho National Laboratory

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http://www.inl.gov

Prepared for the
U.S. Department of Energy

Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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SUMMARY

A fire occurred in a CEM MARS 6 analytical microwave during microwave-assisted, acid digestion of a soil sample in a Research and Development (R&D) laboratory. The soil sample had been pretreated at 550ºC for approximately 4 hours in a furnace prior to digesting in the microwave. Less than 1-gram aliquots of the pretreated soil sample were placed into separate microwave reaction vessels along with an acid mixture used for digestion. The microwave was used to facilitate digestion of the samples. Microwave reaction vessels were loaded with acid and soil samples, sealed in cassettes, and the cassettes placed onto a microwave carousel. A temperature probe that senses the reaction temperature during the run was inserted into the Reference Cell thermowell. The contents of the Reference Cell were identical to the contents of the other reaction cells. The researcher initiated the run and then left the laboratory to perform other work. Upon returning to check the digestion run, flames and smoke were observed originating from the microwave. The researcher exited the lab, immediate emergency actions were taken, and the local fire department responded and extinguished the fire. No personnel were injured, and no facility structural damage occurred. The affected microwave is a total loss. The fire investigation revealed the most credible cause of the fire is an incompatible temperature probe that did not accurately measure temperature of the liquid phase of the Reference sample.

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FIGURES

TABLES

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ACRONYMS

a.m.	Ante Meridiem
^{o}	Degree
^{o}C	Degree Celsius
GB	Gigabyte
GF	Gauge factor
HNO_3	Nitric Acid
HF	Hydrofluoric Acid
in-lb	Inch Pound
INL	Idaho National Laboratory
IR	Infrared
MHz	Megahertz
Micro SD	Micro Secure Digital
mL	Milliliter
Mins	Minutes
P/N	Part Number
PSI	Pounds per Square Inch
PVC	Polyvinyl Chloride
W	Watt

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CEM MARS 6 Microwave Incident

1. INTRODUCTION

CEM Corporation, based in Matthews, North Carolina, is an international laboratory instrument and scientific methods development company that has designed and developed laboratory instrumentation and scientific methods (both microwave-based and non-microwave technologies) for more than 40 years. CEM microwaves are used by major companies, research institutes, and universities around the world for sample digestion as well as materials synthesis. With more than 40-years of experience developing microwave products the engineering design, features, and capabilities of the CEM MARS 6 microwave (Figure 1) reflects CEMs industry-leading position.

Figure 1. CEM MARS 6 stock photo.

The CEM EasyPrep (Figure 2) carousel assembly can hold up to 12 Teflon® sample digestion cells having an internal volume of 100 ml each. The EasyPrep cells are rated to 300°C at 800 psi and are compatible for use with concentrated mineral acids and bases as well as solvents. Under those conditions the most recalcitrant of materials are rapidly attacked by strong mineral acids/caustics and rendered into soluble forms. The EasyPrep vessel assembly (Figure 3) consists of a Teflon® vessel which is the main reaction vessel. A composite, thermally-resistant, structurally-rigid liner slips over the reaction vessel to lend it structural rigidity and to contain the thermal energy within the reaction vessel. Each vessel is capped with a Teflon® lid. Each lid has a steel support ring to prevent the seal from extruding under pressure. The vessel, support sleeve, and lid sit within a cassette module made of grey glass-filled polypropylene. Compressive sealing is applied to the lid of the vessel through use of a glass-filled Ultem compression lid and gland nut. Lids are sealed using 84 in-lb torque from a factory-supplied, calibrated torque wrench. The EasyPrep ensemble also uses a Reference cell which is equipped with a more substantial lid and a thermal well. The thermocouple probe, used for measuring process temperature during the run, inserts into the thermal well. The CEM MARS 5 and early MARS 6 units utilize both thermocouple and infra-red sensors to monitor reaction vessel temperature. Later versions of the MARS 6 utilize in-situ wireless technology for temperature sensing in each vessel during operation.

Figure 2. EasyPrep stock photo.

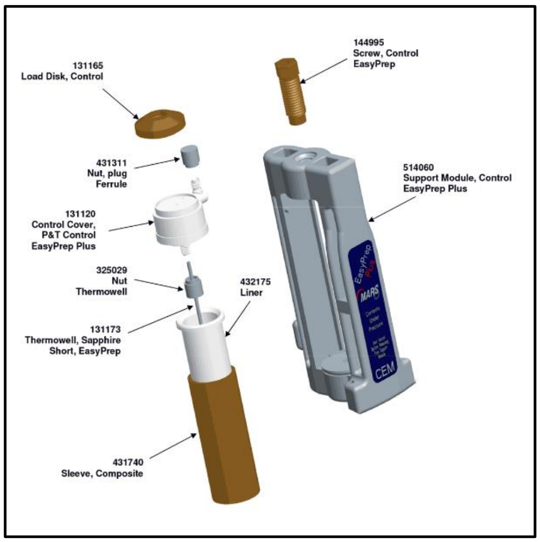

Figure 3. EasyPrep assembly stock photo.

Microwave digestion is not without its inherent hazards, and after 40 years of business CEM has equipped the MARS 6 with multiple mitigation technologies. The ReactiGuard feature senses acoustic and pressure fluctuations in the microwave cavity and will halt the run if a rupture occurs. The on-board software can be used to toggle the ReactiGuard ON/OFF. The MARS 5 and MARS 6 units support both thermocouple and pressure sensing devices to monitor and control the process. Exhaust line acid/organic vapor sensors are optional equipment that can be used and are fully supported by MARS 5 and MARS 6, to shut down a run in the event of an eruption or over-pressurization event.

Microwave heating is also known as dipole heating. Materials having a dipole moment are susceptible to microwave heating. The CEM MARS units employ an 1800-watt magnetron cycling at 2450 MHz which is an ideally suited wavelength to interact with the water dipole, but many chemicals having a dipole will be activated at 2450 MHz. Therefore, the materials of construction for all components going into the microwave cavity are either microwave transparent, or non-absorptive. Only the sample contained in the reaction vessel will be heated by the microwave. Heat generated by activation of the sample is contained by the thermal composite sleeve and the 84 in-lb compression supplied to the lid assembly. Each lid is equipped with an over-pressure release port which will vent over-pressure and then reseal. The IR-sensor and thermocouple monitor process temperature, and the ReactiGuard (if enabled) and exhaust sensor (if enabled) can halt the process if a release occurs. There are other safety systems which will activate and shut down a run if something goes awry. For example, if the carousel stops rotating, or if any of the electronics or fans stop functioning then the run will shut down. If any part of the cell assembly is not routinely cleaned and foreign, microwave-absorbing materials are introduced to the cavity and stuck to the outside of the cell assembly, then those foreign materials become spots which heat, weaken, and damage the vessel assembly. If a rupture occurs during the run that is not detected by the ReactiGuard (if enabled) then rapid heating of the cassette assemblies and carousel can occur causing damage, and in more severe cases causing fire. If incompatible components, thermocouples, sensors, etc., are used then the probability of damage and fire significantly increases. If a thermocouple serial number and/or gauge factor are incorrectly entered, the software then inaccurately reports temperature readings and the microwave can either under-power the run, or worse, over-power a run leading to instrument damage and possible fire.

On the morning of Wednesday November 6, 2019, a laboratory researcher working in INL Research Center laboratoryA-1 started a microwave digestion run designed to digest soil samples in a 50:50 matrix consisting of concentrated nitric (HNO_3) and hydrofluoric acids (HF). Five CEM EasyPrep Teflon® sample vessels (100 ml each) were loaded with ~0.5–0.6 g soil and 4 ml each of conc. HNO_3 and conc. HF. Soil samples had been thermally pre-treated at 550°C for ~4 hr. The Reference cell was included in the run and contained an identical soil/acid composition. Table 1 below gives the power sequence for the microwave program used to digest the samples in the CEM MARS 6 analytical microwave as reported by the researcher. From the table it can be surmised that the overall run time was approximately 160 min (approximately 2 hr 40 min).

Table 1. CEM MARS 6 powers sequence used for soil samples.

Step #	Power (W)	Ramp Time (mins)	Hold Time (mins)	Target Temperature (°C)
1	800	20	20	200
2	400	20	0	25
3	800	20	20	200
4	400	20	0	25
5	800	20	20	200

From post-event evidence it has been determined that the arrangement of the 5 cassettes onto the microwave carousel prior to run start was as is depicted in Figure 4 below. The carousel cleat was in the front/start position, the Reference cell (labelled) was positioned to the front right prior to run start, and the remaining 4 sample cells (in clockwise order) were in the #10, #7, #5, and #3 sample positions, respectively. The thermocouple was inserted into the Reference cell and the digestion sequence was initiated. The researcher then left the lab to attend to other work.

At approximately 10:10–10:20 a.m. the researcher returned to the lab and noted the laboratory space was filled with smoke and the microwave was on fire and emitting active flames. The researcher immediately sought assistance from other research staff. Help was secured and the A-1 laboratory manager pulled the fire alarm; initiating INLs emergency response sequence. The Idaho Falls Fire Department responded and extinguished the flames. In this event there were no injuries to personnel, nor was there any damage to the laboratory facilities. The entirety of damage was contained to the CEM MARS 6 microwave which was a total loss (see Figure 5 and Figure 6). An incident investigation team was formed consisting of INL Fire Safety staff and INL research staff familiar with the CEM MARS 5/6 microwaves and microwave-mediated digestion operations in a research laboratory setting.

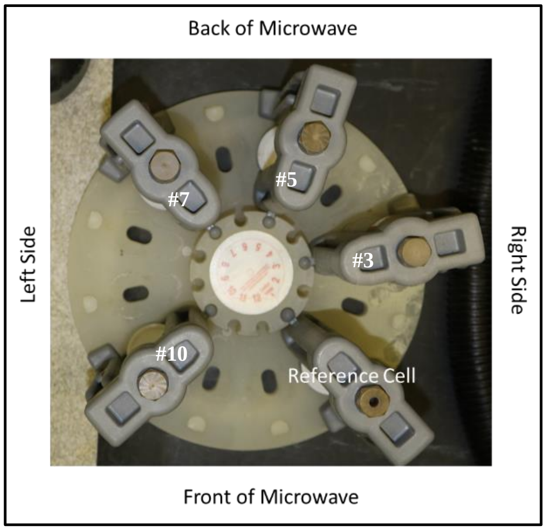

Figure 4. Top down view of carousel and sample cassette configuration as loaded prior to start.

Figure 5. Front left view of microwave.

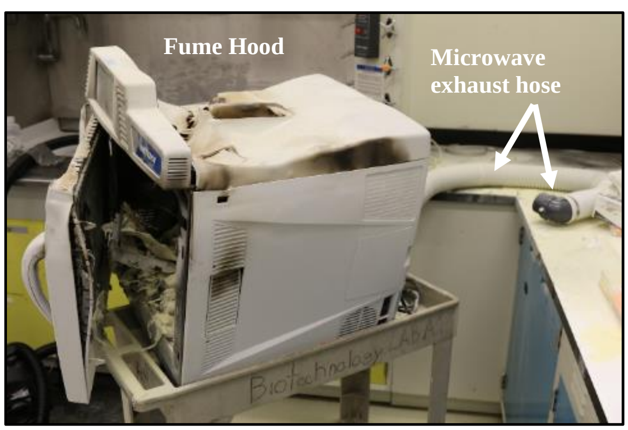

Figure 6. Front right view of microwave.

Immediately after the incident the affected microwave unit was left “as-is” where the Idaho Falls Fire Department firefighters had left it (removed from the bench and placed onto a cart) after extinguishing the fire. The unit, cart, and affected area were cordoned off, and the laboratory door was posted with a limited access sign.

On Wednesday November 20, 2019, the INL incident investigation team entered the laboratory. The team’s objective was to assess the unit and gather evidence which would indicate where the fire started, and what factors contributed to the combustion event. This report provides notes, photographic evidence, and data obtained from investigation activities surrounding the incident.

Figure 5 and Figure 6 indicate the intensity of the heat generated by the fire and the consequent damage to the unit. In Figure 6 the microwave’s white exhaust hose (background, right) was detached from the back of the unit and left on the laboratory bench by the fire suppression team. Yellow fire extinguishing media coated the local bench tops and the microwave unit. The inside of the microwave was amply covered in extinguisher media. The microwave exhaust hose attaches to the rear of the microwave via the grey 90˚ PVC fitting seen in Figure 6. The exhaust hose was routed to the adjacent fume hood seen in the background of the Figure 6 photograph (background, left). From Figure 7 and Figure 8 it is seen that smoke was being actively vented to the fume hood during the microwave run as evidenced from the soot marks where the exhaust hose vented to the hood, and the presence of soot in the exhaust line. Additional evidence obtained by examining soot patterns and melting on exterior panels and vents of the microwave point to the fire occurring in the microwave cavity. The strongest evidence showing the most intensive heating and damage due to fire was found within the microwave cavity itself.

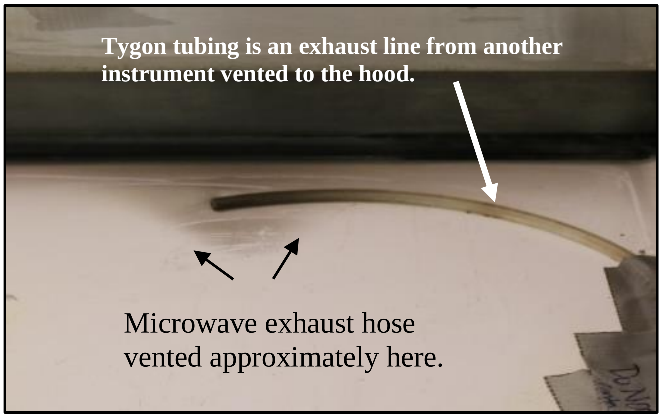

Figure 7. Soot marks in hood where microwave exhaust line (not shown) was located.

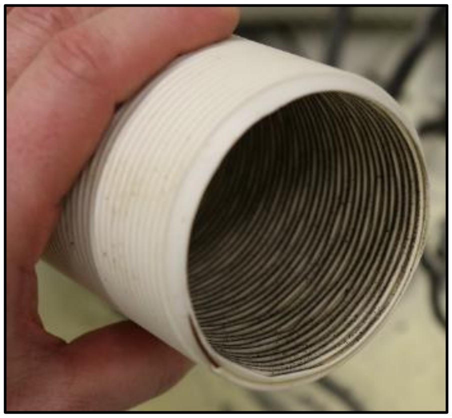

Figure 8. Soot in the exhaust line.

Figure 9 and Figure 10 show what greeted the investigation team when the damaged microwave door was opened. The microwave contents were covered in finely-divided, yellow extinguishing media. It was noted the bulk of the material consisted of unrecognizable, melted polymeric materials interspersed with intact fiber-reinforced composite sleeves which serve as a thermal barrier between the Teflon® sample vessels and the grey, glass-filled polypropylene sample cassettes. The polymeric door molding was melted and had fallen into the microwave cavity. The steel microwave cavity walls were soot blackened and covered with extinguishing media. Teflon® sample vessels were mostly intact where they were protected by the composite sleeves, but no liquids remained, and as will be seen later only one sample had an intact lid; all other sample vessel lids were off including the lid to the Reference cell. In only two instances could lids be confirmed as belonging to their original sample cells (i.e., the Reference cell, and cell #10). No glass-filled polypropylene sample cassette remained intact. Only cassette bases remained and were melted to the top of the carousel.

Figure 9. First look at microwave contents.

Figure 10. Second look at microwave contents.

In order to make sense of the key pieces and to facilitate recovery of vital information the entire monolithic mass of melted polymer was lifted intact out of the microwave and placed into the adjacent chemical fume hood as is shown in Figure 11. The orientation was kept intact. Note from Figure 12 below when the mass was removed from the microwave the carousel cleat was determined to be facing towards the back of the microwave; 180˚ opposite the start/finish cleat position. That finding indicated the run cycle did not finish, and the microwave carousel stopped rotating sometime during the run.

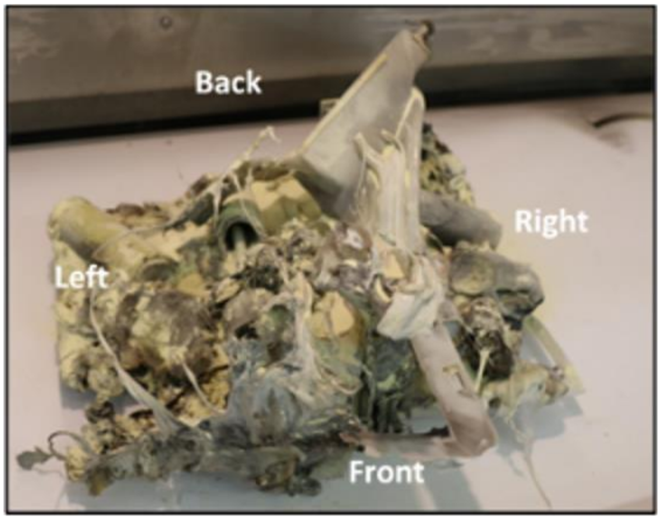

Figure 11. Microwave contents lifted intact and placed into the fume hood.

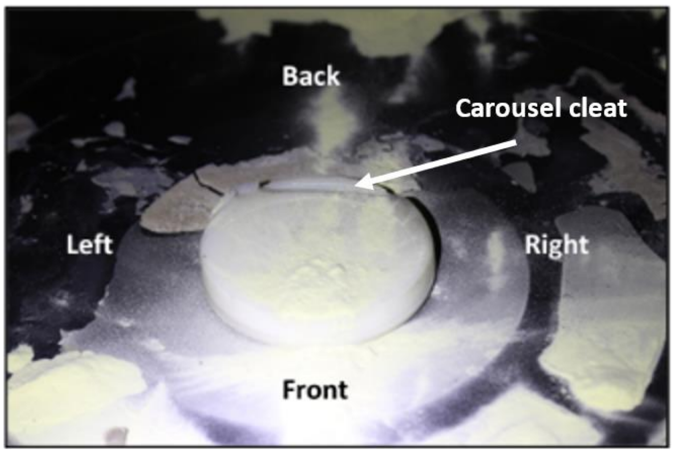

Figure 12. Microwave carousel cleat. The cleat is pointed at the back of the microwave; 180 degrees off the start/finish position.

Determining the position of the carousel cleat allowed for assigning the proper orientation of the different sample cassettes, sample vessels, and associated vessel components. Figure 13 shows a top-down view of an intact carousel and sample cassettes as they would have appeared in the microwave when the carousel stopped in its current position. By contrast, Figure 14 shows the microwave contents "as-is" with the carousel cleat in the 180˚ position, and sample assignments given in the picture. From Figure 14 it is seen that all cassettes have been melted down to the base which sits on the carousel. The Reference cell is at the back of the microwave with the top of the cell tipped towards the front of the microwave (Figure 16). The lid of the Reference cell is off and is located several inches in front of the Reference cell towards the front of the microwave (Figure 16). Sample cell #10 is at the very back right-hand side of the oven and the top has tipped toward the back right-hand corner (Figure 14 and Figure 15). The lid on Sample cell #10 is intact and has become melted (fused) to the body of the cell. Sample cell #7 has tipped inwards towards the carousel hub with the bottom of the sample cell and composite sleeve touching the right-hand side of the microwave cavity (Figure 14 and Figure 15). Cell #7 is missing its lid. Lids cannot be assigned to either cells #5, or #7. One sample lid was found on the floor; it cannot be definitively assigned to #5, #7, or #3. Sample cell #5 has also tipped inwards towards the carousel hub and the sample cell appears to be pointed towards the back wall of the microwave cavity (Figure 14 and Figure 15). Sample cell #3 has tipped towards the back left-hand corner of the microwave and is missing its lid (Figure 16); however, the presence of the black steel retainer ring may indicate the Teflon® #3 sample lid has melted. Figure 15 and Figure 16 show sample cell identifications and orientations as viewed from the right side (Figure 15), and from the left side (Figure 16).

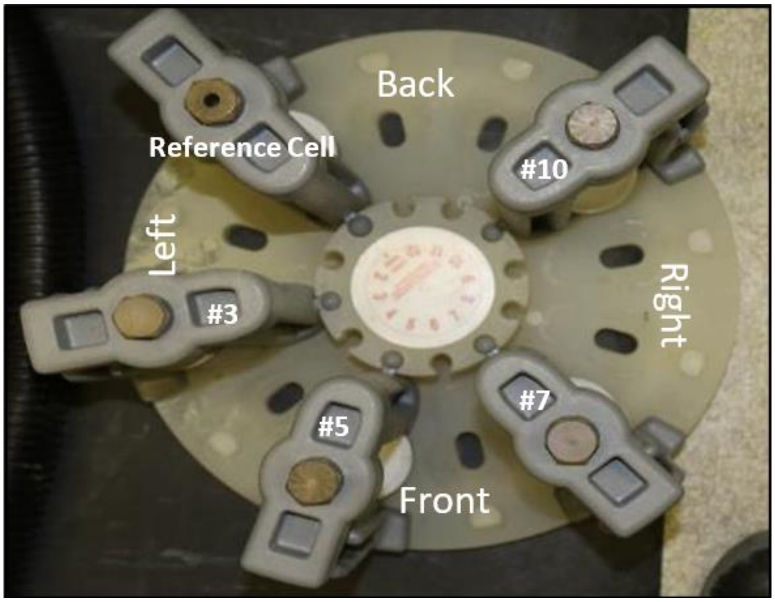

Figure 13. Top down view of carousel as it stopped.

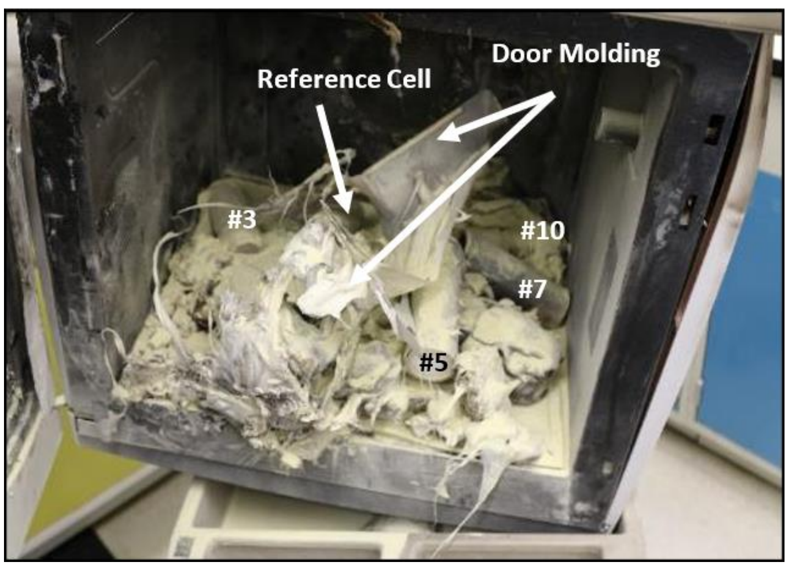

Figure 14. Sample cells identified in “as-is” condition.

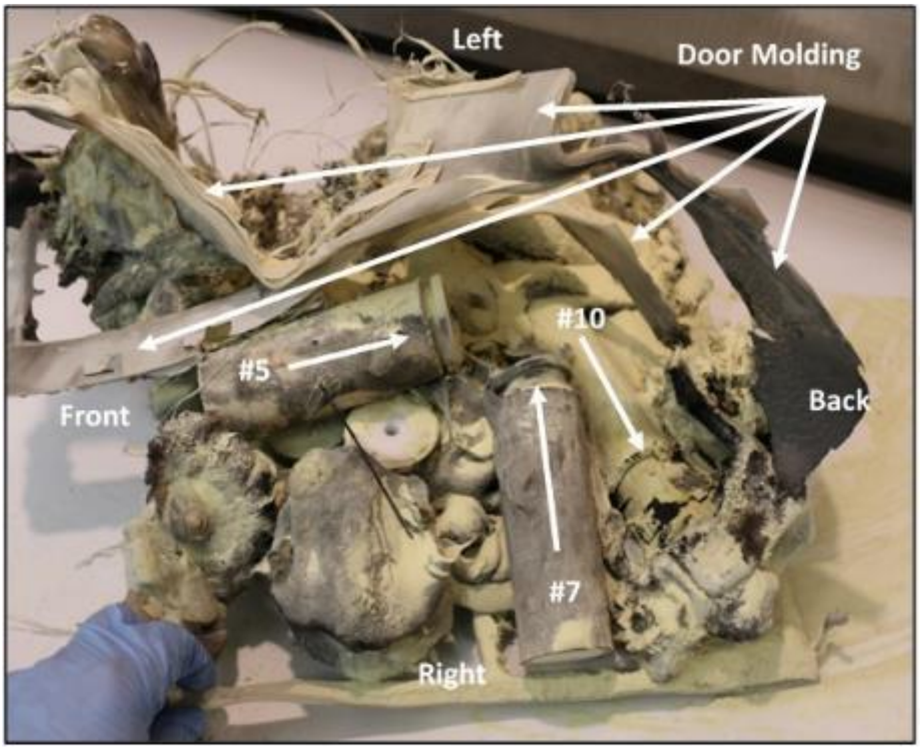

Figure 15. Sample cells identified in “as-is” condition. View from the right-hand side.

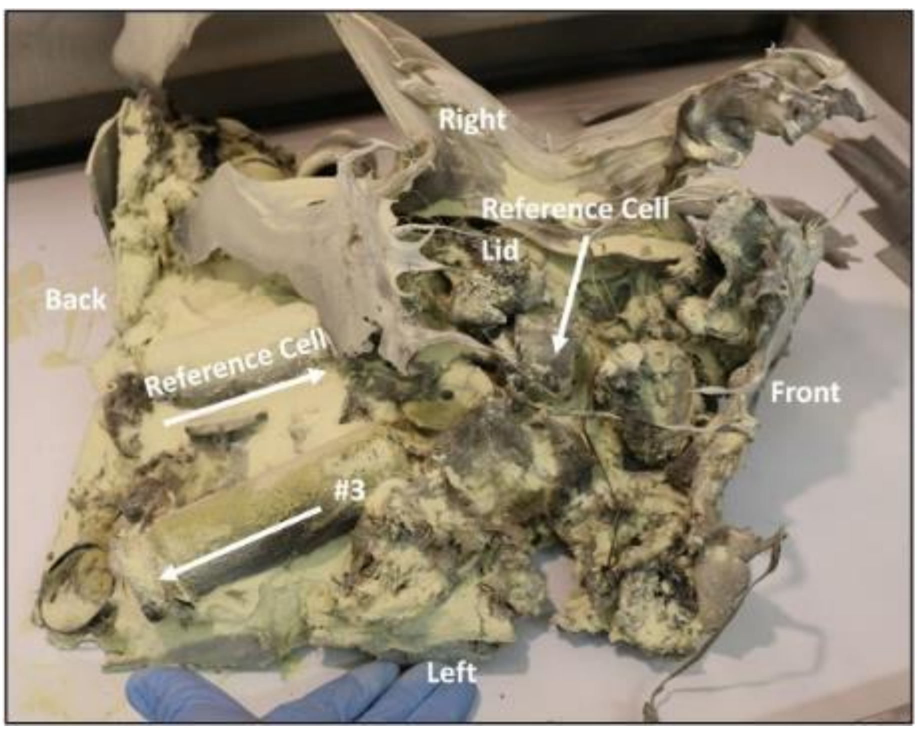

Figure 16. Sample cells identified in “as-is” condition. View from the left-hand side.

Work was then undertaken to remove non-essential components, such as the melted door molding, so that the contents of the carousel could be cleaned of extinguishing media, photographed, and retrieved for examination. A Shop Vac equipped with a high efficiency collection bag and filter were used to remove the extinguishing media. Care was taken not to vacuum up any vital component or to disturb the arrangement. Some of the glass fiber from the glass-filled polypropylene sample cassettes was inadvertently vacuumed up but deemed to be irrelevant to the task. A stream of forced air was also employed in delicate spots to blow the extinguishing media away from the mass. The task was conducted in the fume hood which captured the fines coming off the mass.

A vital piece of information recovered during the operation was the location and status of the Reference cell, lid, and the thermocouple. Figure 17 shows a close-up of the Reference cell lid and the thermocouple coming out of the lid.

The thermocouple was broken into three sections. One section was found to be intact and still inserted into the Reference cell lid (Figure 17). The external coating on that section of the thermocouple was relatively unscathed and ran between the cell lid and a mass of fibrous plastic (upper right of Figure 17) which appeared to be the melted remains of the glass-filled Ultem load disk, top of the Reference sample cassette, and the glass-filled Ultem compression gland nut.

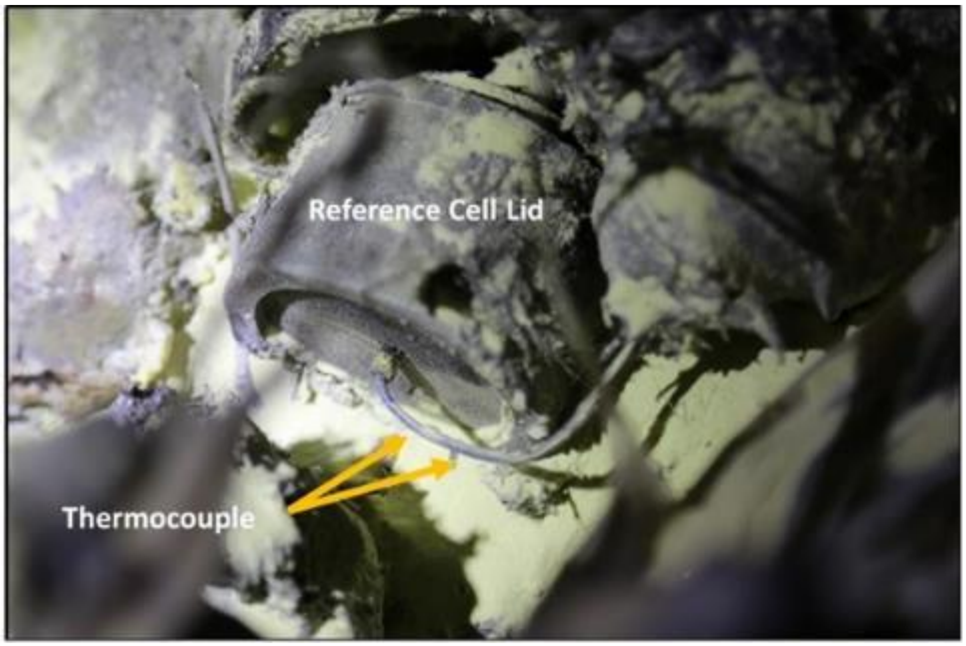

Figure 17. Reference cell lid and thermocouple coming out of the cell.

The second section of the thermocouple was found sitting on top of a melted mass of plastic located between Sample cell #5 and Sample cell #7 (Figure 18). That section was broken at both ends with the central fiber exposed at both ends, and the coating was more severely damaged from exposure to direct flame (charring). From appearances, the middle section of the thermocouple appeared to have fallen to its present location much later in the event cycle.

Figure 18. Middle section of the thermocouple.

The third section of the thermocouple was still attached to the fixture in the ceiling of the microwave cavity (Figure 19), and that section of thermocouple was extremely damaged from heat, showing severe melting of the coating and central fiber. The serial number and other identifying marks on the thermocouple were damaged beyond recovery. The only definitive evidence showing what type of thermocouple was used is the blue coating on the section of thermocouple coming out of the Reference cell lid.


Figure 19. Thermocouple section dangling from the ceiling fixture.

Figure 20 clearly illustrates the blue color of the coating on the thermocouple jacket exiting the Reference cell lid. Additional supporting evidence showing the type of thermocouple used during the run can be obtained from manufacturer literature and product containers thermocouples arrive in.

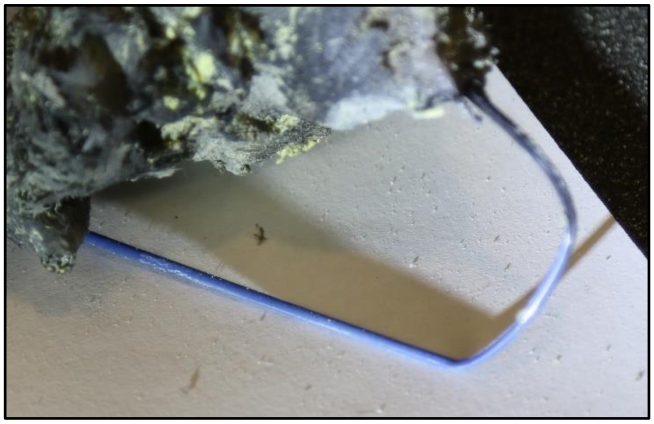

Figure 20. Blue jacketing material on thermocouple exiting the Reference cell lid.

Figure 21 shows examples of the white MTS-300 series thermocouple probes in clear plastic product containers in which the probes arrive from the manufacturer. The boxes are labelled with the part#, serial #, gauge factor, and temperature range. MARS 6 temperature probes are 20 inches long and either all white (MTS-300 P/N 314355) or white on top with a black coated section near the sensing tip (MTS-300 High Temp P/N 281461). MARS 5 probes are 16 inches long and either blue topped with a blue tip (RTP-300+ P/N 314305), or blue topped with a black tip (RTP-300+ High Temperature P/N 314306).

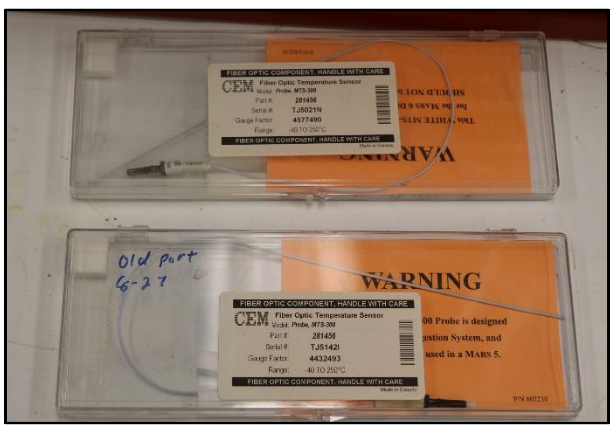

Figure 21. White MTS-300 series thermocouple probes compatible with CEM MARS 6.

The different MARS 5 and MARS 6 temperature probes are displayed in the CEM Fiber Optic Probe Guide shown in Figure 22. Due to the 4-inch length difference between the blue MARS 5 probes and the white MARS 6 probes the manufacturer has supplied an orange warning card in each product container expressing the incompatibility between different MARS probes and their uses in different MARS microwaves. When a 16 in. MARS 5 probe is used in the MARS 6 microwave the shortness of the thermocouple allows it to only be inserted a short distance into the Reference cell's thermowell. The probe tip, which is the active temperature sensing portion of the thermocouple, does not extend into the liquid portion of the sample; thus, leading to measurement of temperature appearing in the vapor phase of the sample which is not an accurate representation of the temperature of the liquid in the sample. Further, even though a MARS 5 blue thermocouple fits within the MARS 6 thermocouple fitting, and the serial# and gauge factor can be programmed into the MARS 6 software, if the gauge factor number is not correctly entered then the probe can report an inaccurate temperature. If a fiber optic temperature probe breaks at any time during the run, then there will be a loss of signal between the probe and the control board. If that happens, the software stops the run immediately (stops applying microwaves) and displays an error message. The software will not allow the run to continue with a broken probe.

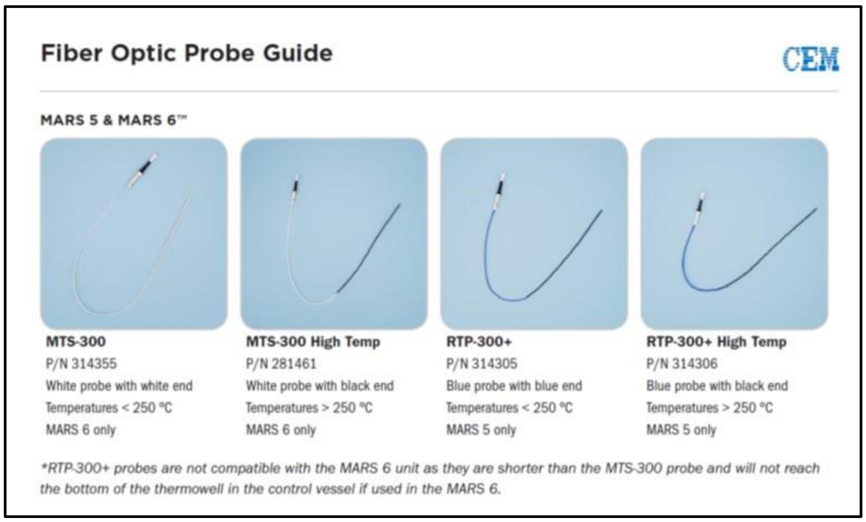

Figure 22. CEM MARS 5/6 Fiber Optic Probe Guide.

Figure 23 shows a thermocouple probe box for a blue CEM MARS 5 probe P/N 314305. The box is empty. Two other thermocouple boxes were located on the lab bench immediately adjacent to where the MARS 6 was located. Both of those boxes contained white MTS-300 style MARS 6 probes.

Figure 23. One of the three thermocouple boxes obtained from laboratory A-1 bench proximal to the microwave and containing supplies.

Figure 24 shows the orange manufacturer-supplied warning label found in the MARS 5 thermocouple box. The Reference cell, lid, and blue thermocouple pieces were carefully collected, photographed, and bagged. There is strong evidence that a blue MARS 5 thermocouple was deployed during the run.

Figure 24. Manufacturer-supplied warning label found in the MARS 5 thermocouple box.

As many of the carousel components were cleaned and retrieved as was deemed practical. Afterwards the carousel was lifted onto the back edge to view the underside of the carousel. Figure 25 shows the underside of the microwave carousel. The story told by the underside of the sample carousel points to the back and back-left side of the carousel having experienced the hottest temperatures as is noted from the considerable amount of melting that took place. The front-right of the carousel is still relatively intact from the underside and the carousel track balls are still rolling and functional. Even though from the top-side the cassettes were melted down to their bases, the front-right under side was relatively unaffected.

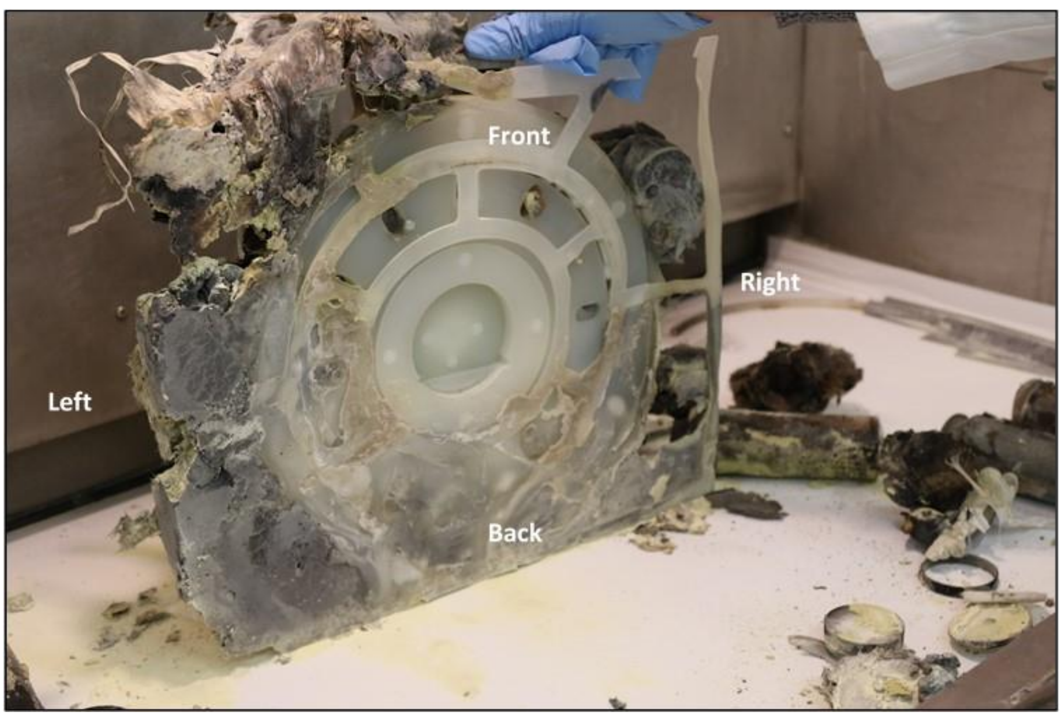

Figure 25. Underside of the carousel.

2. MARS 6 RUNTIME DATA FROM THE ON-BOARD MICRO-SD CARD

The MARS 6 analytical microwave is equipped with an on-board computer. Run sequences, programs, and real-time run data are stored on a Micro-SD memory card. Even though the plastic around the card was found to suffer from a minor amount of melting due to the heat of the fire, the on-board computer module and Micro-SD card appeared to be undamaged and intact (Figure 23).

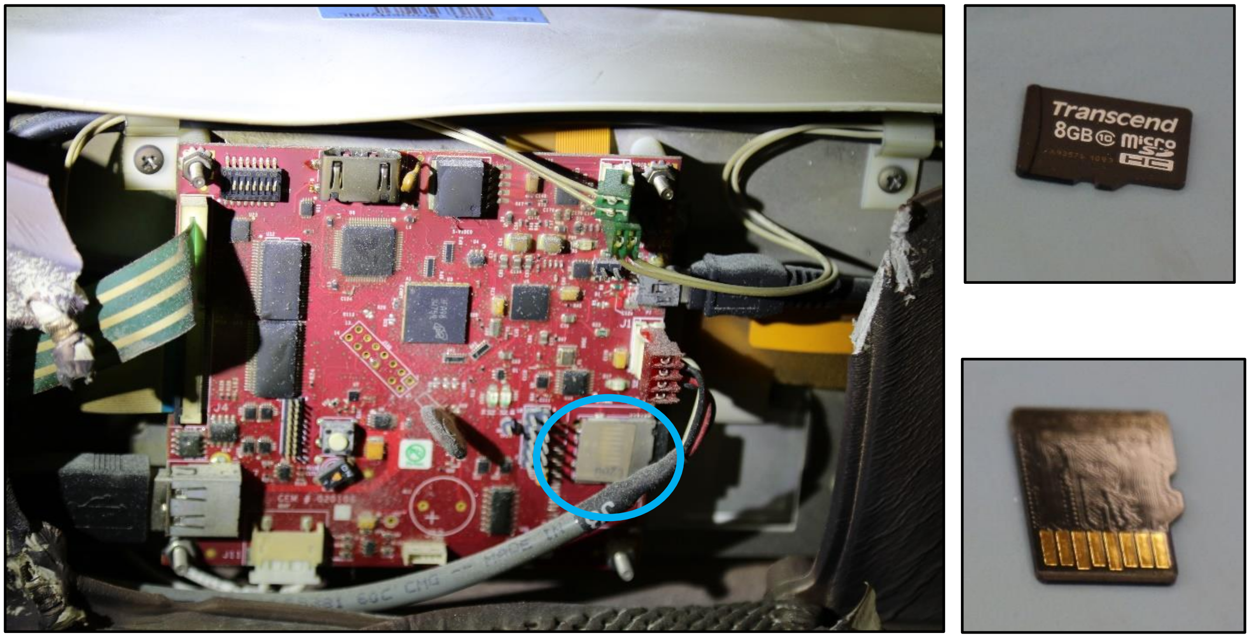

Figure 26. On-board computer module and Micro-SD card (encircled in blue). Top right: 8 GB Micro-SD card. Bottom right: Flip side of Micro-SD card showing the leads are not damaged.

The plastic cover over the computer module was removed and the Micro-SD card was retrieved with care and following manufacturer's instructions. The memory card was packaged to prevent damage during shipping (as instructed by the manufacturer) and sent to CEM Corporation for analysis.

Runtime data retrieved from the memory card indicated the method running during the last run was the "Mann Soil" method communicated by the research staff. However, the run sequence of the saved run was slightly different than that reported by the research staff and shown in Table 1 above. The actual saved Mann Soil run as reported from the Micro-SD card showed a 60-minute run, where initial power = 1800 W with a ramp over 30 minutes from ambient to 170°C. The run was then held at 170°C for 30 minutes. The Micro-SD card data also reported the on-board software version running at the time was v1.26. The latest MARS 6 software is v1.51. CEM provided a list of software updates to the investigation team that had been released since v1.26.

The Micro-SD card also reported the thermocouple GF = 448-9482, and the temperature probe programmed into the instrument was assigned S/N TJ5009N, which cross-references to an all-white MTS-300 MARS 6 probe. The thermocouple probe used in the final run was a blue probe. Power-Temperature-Time data from the final run was retrieved from the memory card and compared to previous Mann Soil runs that were successfully completed. Runtime data show the power-temperature ramp for the final run proceeded consistently for ~22 minutes. At Time = 22 min. the temperature rise slows significantly and levels at ~141°C. In response to the temperature lag the microwave went to full power ~1800 watts for ~14.5 minutes, but the temperature only went to ~149°C. The run was aborted at 40.1 minutes by the ReactiGuard. The runtime data showing power and temperature as a function of time and compared against a successful earlier run are shown in the graphs given in Figure 27 and Figure 28.

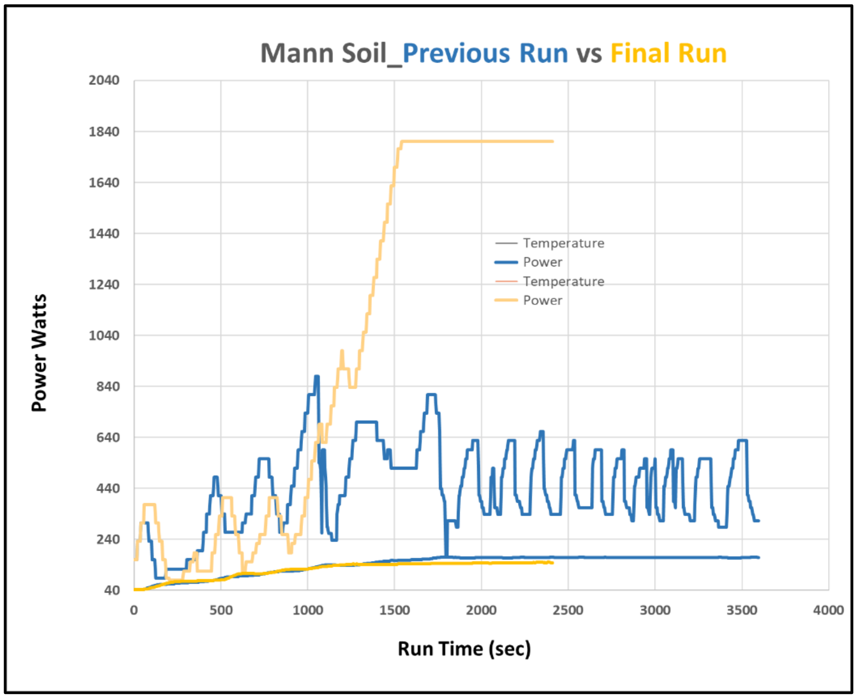

Figure 27. Power vs. run time in seconds. Previous successful run data (blue) compared to the final run data (yellow). The final run ramps to full power (1840 watts) after approximately 22 minutes and runs for another ~14.5 minutes before the run is terminated.

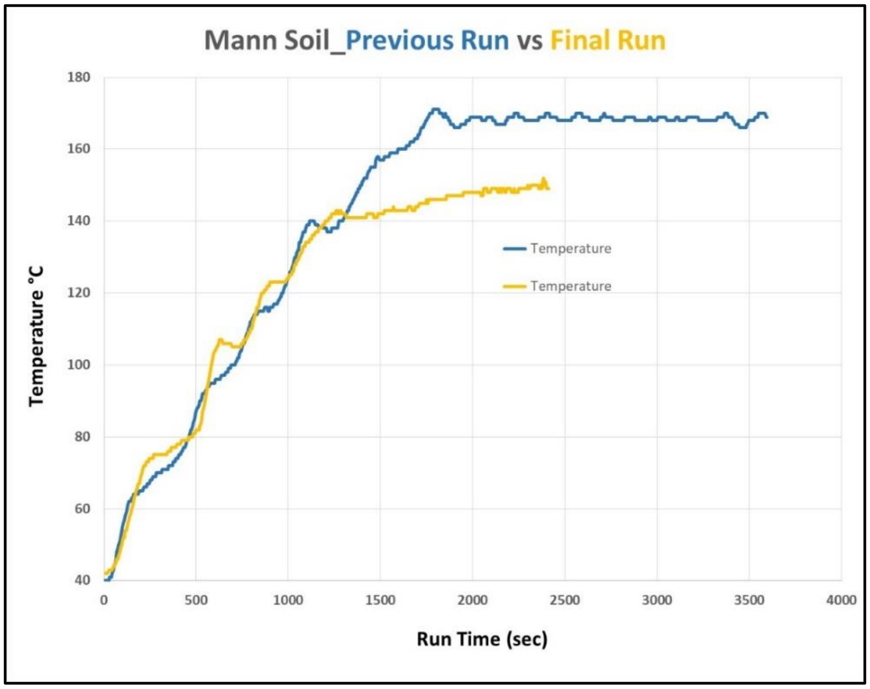

Figure 28. Temperature vs time (sec) for a previous run (blue) and the final run (yellow). The probe used for the final run did not come to temperature causing the microwave to go into a full power mode to bring temperature to the set-point.

3. CAUSE OF FIRE AND SEQUENCE OF EVENTS

Evidence gathered by the investigation team indicates a blue MARS 5 RTP-300+ thermocouple was used for the final microwave run. Additionally, the gauge factor for the new probe and the serial number of the new probe were not properly programmed into the microwave. The microwave had not been re-programmed and the data for the previous probe (an all-white MTS-300 probe) was still in the memory buffer. Regardless, even if the proper GF and the S/N had been used the blue MARS 5 probe is too short to be used in the taller MARS 6 microwave and can lead to inaccurate temperature sensing because the short probe senses the headspace of the Reference cell, and not the liquid phase. The microwave operator stated the probe had been changed out the morning of the fire due to the old probe being taken out of service. The operator searched through the spare probes and tried several. After trying several probes that would not allow the run to initiate, the researcher found one that apparently would allow the run to start, and then proceeded to initiate the run. Use of an incompatible probe does not guarantee an event, but rather significantly increases the probability of an untoward event occurring. After the run initiated the researcher left the lab.

The torque wrench used to compress sample lids was tested. Even though the wrench was set at 83 in.-lb, and the manufacturer specifies 84 in.-lb, the wrench at its current setting was supplying 81.56 in-lb torque which is 2.9% low, but still well within the manufacturer's tolerances for the torque wrench and force applied to the MARS 5/6 sample lids. Even though the lids were tight, the blue MARS 5 probe reported an incorrect temperature and over-powered the run causing loss of acid contents into the microwave cavity. The temperature probe did not break during the run. Microwaves kept pouring into the overheated and venting sample containers. The run eventually stopped in the 180° position and intensive heat damage could be seen to sample cells #10, the Reference cell, and cell #3. It is unknown which sample cell or cells vented first, and it really does not matter because the results are the same, but #10, #3, and the Reference cell are most strongly suspected. Sample cell #10's lid had undergone such intense heat that the sample cell lid was fused to the cell body and the composite thermal barrier sleeve was scorched (Figure 29).

Figure 29. Scorched composite thermal sleeve surrounding Sample cell #10.

When cell #10's lid was pried off it was noted that a solid mass of material remained in the vessel which appeared to be undigested or partially digested, consolidated, soil sample; an indication of loss of acid early in the run cycle. Loss of contents caused for very hot concentrated acid (HNO_3/HF) to exit the sample vessel(s) at >800 psi. If the blast were directed at the cassette body, the glass-filled polyethylene would have melted. Fire was caused when microwave transparent materials became microwave absorbing and the temperature sensor failed to shut the microwave off, and microwaves continued to rapidly over-heat cassette polymers. The primary material combusted was the grey, glass-filled polyethylene cassettes. The ReactiGuard failed to sense a vessel rupture and did not shut the microwave off until active smoke and flame were present. Microwaves rapidly over-heated cassette polymers causing loss of structural integrity and venting of vessels. Even though polypropylene is somewhat chemical/acid resistant, very little can handle >200°C concentrated acid blasting out at >800 psi. Fire was caused by the temperature probe failing to accurately sense temperature and causing significant heating of the acid-splashed cassette polymers which caught fire. Fire and heat were most intense at the back of the microwave, melting polymers, scorching thermal shielding, and melting the main fan shroud in the back of the microwave.

4. RECOMMENDATIONS

The investigation team has obtained evidence pointing towards significant contributing factors outside of an incompatible blue MARS 5 thermocouple probe (16-in.) used in a MARS 6 device.

- Incorrect serial number and Gauge Factor programmed into the instrument.
- Unattended run. Acid fume sensor (optional, but not purchased) on exhaust line not used.
- MARS 5 thermocouple kept with MARS 6 spare probes and MARS 6 device.
- MARS 5 thermocouple fits into the MARS 6 connector and will not ERROR OUT.
- Thermocouple product boxes and labeling are similar. Probes removed from boxes were not returned to their original boxes matching their S/N and GF on the front label of the box.
- At least 3 different colored warning labels containing 4 different warnings in probe boxes.
- A 100-page user manual that mentions nothing about MARS 5 and MARS 6 part incompatibility.
- No known product recalls or other warnings for incompatible probes.
- CEM database showed the microwave was owned by a company located in North Carolina. INL Procurement using 3rd party vendors, who gets the updates?
- Cognizants listed in the CEM database for this instrument were INL staff present at the time of installation, but who are not the present microwave owners/operators.
- User Manual revisions and critical software revisions not communicated to the microwave users. Research staff not notified of updates. It was noted that in one software update the maximum temperature for the EasyPrep cassettes had been reduced by the manufacturer. Further, a software revision had been released which limited maximum power to <1600 watts, and if the microwave went into a full 1600-watt power run the microwave would terminate the run if the thermocouple failed to register a corresponding temperature rise after 5 minutes.
- Storage of incompatible parts with compatible parts.
- Safety equipment listed as "optional" by the manufacturer. Equipment not purchased.
- New probe (safety-critical equipment) installed in a device without investigating specifics regarding the thermocouple.
- No evidence of routine maintenance of the torque wrench.

- Over-reliance on equipment, the manufacturer, on past successful runs, and on personal knowledge/skills.
- Sign blindness. Bright orange WARNING label in the probe box.
- Conditioning/culture that enables us to ignore instructions and warning labels.

In anticipation of completion of this assignment there are some MARS best practices that can be offered to the CEM MARS 5/6 user community at INL. Note these suggestions are very much repetition of common best practices already offered/practiced by the manufacturer and other experienced users.

1. Check temperature probe performance and compatibility with the instrument and intended conditions.
2. Check probe S/N and Gauge Factors to ensure they are properly encoded in the on-board software.
3. Keep MARS 5 and MARS 6 probes separate.
4. 84 in-lb is the torque recommended by the manufacturer to seal the EasyPrep assemblies. Proper torque is necessary to prevent venting scenarios and is a function of a properly operating torque wrench.
5. Check the on-board software to ensure safety features like the cavity acoustic/pressure sensor are enabled in the ON state.
6. Unattended experiments should employ exhaust acid/organic vapor sensors.
7. Routinely and frequently wipe and clean the in-cavity IR sensors.
8. Routinely and frequently check lids, seals, cassettes, and sample vessels for warping, cracking, damaged, and ill-fitted lids and sealing surfaces.
9. Routinely and frequently clean all cassette and sample container components to keep them microwave transparent.
10. Follow manufacturer's recommendations for ensuring your MARS is calibrated and delivering the power expected.
11. Get in the habit of performing a "pre-flight" checklist prior to every run.
12. Frequently consult with the manufacture to obtain the latest software/firmware revisions and to be aware of any product safety issues or recalls.