National Transportation Safety Board
Office of Research and Engineering
Washington, DC 20594

PLD23LR002

MATERIALS LABORATORY

Factual Report 23-072

October 2, 2023

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A. ACCIDENT INFORMATION

Location: West Reading, Pennsylvania
Date: March 24, 2023
Vehicle: UGI Gas Distribution Pipeline System
Investigator: Kim West, RPH-20

B. COMPONENTS EXAMINED

Gas distribution system components near the intersection of S. 2nd Ave and Cherry St., West Reading, PA:

Aldyl A service tee, retired from service, with longitudinal split in tower (cap and part of insert missing). Formerly providing gas service to 17 S. 2nd Ave (Lengths of distribution piping and service lines still attached).

Pipe cutter from retired service tee.

O-ring.

Service tee installed in 2021, adjacent to and part of same assembly as preceding Aldyl A tee, providing gas service to 17 S. 2nd Ave at the time of the incident (Lengths of distribution piping and service lines still attached).

Sister (exemplar) Aldyl A tee, servicing adjacent building at 77 S. 2nd Ave (Lengths of distribution piping and service line still attached).

Approximate 94-inch length of plastic 1-inch gas service line, with riser assembly and meter connection fitting.

Marker balls, one from 17 S. 2nd Ave service tee site and one exemplar.

Section of buried 3-1/2 inch nominal pipe size (NPS) steel pipe used to convey steam, approximately 45-5/8 inch in length.

Approximate 31-inch length of 2-inch steel distribution pipe with 1-1/4-inch plastic Aldyl A distribution pipe inside.

1.0 Additional components received (not examined)

Sample of white powder surrounding buried steam pipe.

Soil samples.

C. EXAMINATION PARTICIPANTS

Group Chair	Donald Kramer, Ph.D. National Transportation Safety Board Washington, DC
Group Member	Kim West, IIC National Transportation Safety Board Washington, DC

Group Member	Sara Lyons National Transportation Safety Board Washington, DC
Group Member	Mark Connors UGI Utilities, Inc. Denver, PA

D. DETAILS OF THE EXAMINATION

Components from and adjacent to a gas distribution system located near the intersection of S. 2nd Ave and Cherry St. in West Reading, PA were sent to the NTSB Materials Laboratory in Washington, DC for examination as shown in figures 1 – 5. The in-service gas main was manufactured from medium density polyethylene (MDPE) by DuPont and marketed under the trade name "Aldyl A". The piping was 1-1/4-inch nominal pipe size (NPS) and was installed in April 1982 along Cherry St. by insertion through an existing 2-inch NPS steel main. Gas service was provided from this main to 17 S. 2nd Ave on the north side of Cherry St. (Palmer building #2) and 77 S. 2nd Ave on the south side of Cherry St. (Palmer building #1) through service tees and service lines to each building. Figure 1 shows the segment of the distribution system providing gas service to 17 S. 2nd Ave and Figure 2 shows the system segment providing service to 77 S. 2nd Ave.

Gas service to 17 S. 2nd Ave was originally supplied through an Aldyl A service tee, as indicated in figure 1. The Aldyl A tee was retired from service in March 2021 and a replacement service tee was installed on the main located approximately 9.45 inch downstream.

The retired tee was recovered with its cap missing, a portion of its insert missing, and a longitudinal fracture in the tower shell (figures 6a and 6b; also see Section 1.0 and figure 7 below for a depiction of the service tee components and definitions). The cap and the upper portion of the insert were not recovered. For convenience clock positions were assigned to the perimeter of the tower, with 12 o'clock assigned to the tee outlet and other clock positions assigned when viewing the tee from top down. Using this nomenclature, the longitudinal fracture was located at approximately 10 o'clock. A cutter and O-ring, shown in figure 3, were recovered separately.

At the time of the accident, gas service to 77 S. 2nd Ave was supplied through the Aldyl A service tee originally installed in 1982, shown in figure 2. The tee was recovered intact with tape around the fitting (for holding in place a protective sleeve on the tee outlet) and without any apparent damage or discrepancies. As seen in figure 1, there was no sleeve on the 17 S. 2nd Ave retired tee outlet.

As shown in figures 6c and 6d, respectively, the tape around the retired tire exhibited a wrinkled and dimpled appearance on the top/south side of the gas main and a cracked and split appearance on the bottom/south side of the main. The tape on the north-facing side of the main, by contrast, did not exhibit any noteworthy features, nor did the tape on the tee for 77 S. 2nd Ave (see figure 7).

A section of a nearby buried steam pipe, used to provide radiant heat to parts of 17 S. 2nd Ave., was submitted to the lab as well (figure 5). The pipe was deformed, corroded, and cracked along its east side as discussed below. A white powder surrounded the pipe at the accident site. The material was identified as predominantly calcium carbonate by an external laboratory at a later date. See the Operations Group Chairman Factual Report for additional details on the identification of the calcium carbonate and for photos and illustrations of the excavation site showing the relative locations of the 17 S. 2nd Ave service tees, the steam pipe, and other assets.

Prior to the group exam, baseline dimensions of the Aldyl A pipe were measured using a caliper and the locations of notable features along the section of gas main with the 17 S. 2nd Ave saddle fittings were recorded with a steel tape to the nearest 0.05 inch. The outer diameter measured between 1.672 inch and 1.688 inch for a nominal outer diameter of 1.680 inch. The wall thickness was measured in one location by caliper as 0.175 inch. The calculated nominal inner diameter was 1.330 inch. The positions of notable features along the main in the vicinity of the 17 S. 2nd Ave service tees are listed in table 1 below:

Table 1. Location of notable features (to the nearest 0.05 inch) along the gas main in the vicinity of the 17 S. 2nd Ave service tees.

Feature description	Linear distance along pipe, inch
Exit from casing at west end	0.0 inch
West end of retired tee saddle	5.0 inch
Centerline of retired tee	6.25 inch
East end of retired tee saddle	7.50 inch
Beginning of shaved pipe OD segment	13.30 inch
West end of 2021 tee saddle	13.60 inch
West edge of 2021 tee undersaddle	14.40 inch
Centerline of 2021 saddle	15.70 inch
East edge of 2021 tee undersaddle	17.65 inch
East end of 2021 tee saddle	17.80 inch
End of shaved pipe OD segment	19.45 inch
Entering casing at east end	21.20 inch

The following sections describe the design and operation of Aldyl A tapping tees, testing and examinations conducted during a group examination held between

June 26, 2023 and June 30, 2023, follow on laboratory work after the group activity, and the findings from external laboratory testing. The 17 S. 2nd Ave service line failed a leak check in the field. The service line and riser (figure 4a) were examined prior to the group activity and the findings are included in Appendix A at the end of this report. The marker balls (figure 4b) were photographed and weighed prior to the group activity but were also examined during the group activity and are included in the group exam section below.

After the group activity, the manufacturer's pipe identification markings were examined on a 31-inch length of the Aldyl A gas main located upstream of the 17 S. 2nd Ave service tees. The pipe was identified ASTM 2513 and the material was identified as PE 2306 AF BE CD. See Appendix B for photos of the material markings and for a table used to decode the material suffixes.

1.0 Design and operation of Aldyl A tapping tees

Design and operation of the Aldyl A tapping tees installed in 1982 are described with the aid of photographs and X-ray computed tomography (CT) scans of the sister tee from 77 S. 2nd Ave, shown in figures 7 and 8. The tees are used to branch off an existing Aldyl A gas main. By design, the tee's saddle is fusion welded to the gas main. Downstream gas service piping and fittings are installed and leak tested per operator/installer procedure. Once a leak-free connection is established, a cutter, located in the tee's tower and guided by a threaded insert (see CT scan in figure 7), is turned using a hex key which lowers the cutter and perforates/taps the pipe. The cutter is then backed out of the hole allowing gas to enter the service line. Finally, the tee is sealed by installing a threaded cap, equipped with a rubber O-ring, onto the top of the tower.

The tower is a two-piece assembly, consisting of an insert and an outer shell. The insert (figure 8a) is made of polyoxymethylene homopolymer (also known as polyacetal or by the DuPont trade name "Delrin"). The inside of the insert is threaded for guiding the cutter (figure 8b) and for anchoring the cap (figure 8c). The outside of the insert contains longitudinal and circumferential ribs. The insert is housed inside a polyethylene shell (figure 8a) that is molded and formed around the insert. The ribs on the insert interlock with corresponding grooves in the shell and allow the insert to resist axial and rotational movements during cutting and capping operations.

2.0 Group examination

The group portion of the examination took place from June 26, 2023 to June 30, 2023. Leak testing of the active service tees was conducted on June 26 with a representative from UGI Utilities in attendance. The active and retired service tees were scanned by X-ray CT on June 27. Disassembly of the retired tee and examination of the steam line took place between June 28 and June 30 with representatives from UGI Utilities and the Pennsylvania Public Utility Commission in

attendance. Investigators from the NTSB Pipeline and Hazardous Materials Division were also in attendance for parts of the examination.

2.1 Leak testing

The active service tees, the 2021 replacement tee for 17 S. 2nd Ave (2021 replacement tee) and the 1982 Aldyl A tee for 77 S. 2nd Ave (sister tee), were both leak-tested. The 2021 replacement tee was separated from the retired tee by cutting the gas main with a rotary pipe cutter. External pipe caps equipped with quarter-turn ball valves and pressure gauges were installed on all open pipe ends. Each assembly was in turn connected to shop air regulated between 53 psig and 54 psig (the pressure at the time of the accident was 53 psig). Flow rate was measured using an analog flow meter with a range between 0.03 SCFH and 43.0 SCFH.

Leak testing of the 2021 replacement tee indicated a leak was present with a measured flow rate of 0.6 SCFH. Soap testing around the saddle revealed bubbles forming on the west end (upstream end) of the tee saddle.

Leak testing of the sister tee did not indicate any flow within the range of the flow meter but the assembly slowly lost pressure over time. The assembly was capped and left to hold pressure for 15 minutes. The pressure dropped from approximately 54.5 psig to 53.0 psig over the interval. The test was repeated twice, first after removing a length of pipe with a longitudinal scratch and second after swapping out one of the pipe caps, with similar results. The pressure dropped approximately 1.5 psi to 2.0 psi over the 15-minute interval.

2.2 X-ray CT scanning

On June 27, the active and retired service tees were transported to NTS Chesapeake in Bel Air, MD for X-ray CT scanning. Notable finding from the CT images were as follows:

1) By design, the cylindrical ribs on the insert (and grooves on the shell) were not symmetrical in cross section. As seen for the sister tee in figure 7, the upper portion of the rib transitioned to the cylindrical barrel at a right angle while the lower portion transitioned to the barrel at an obtuse angle.

2) The top three cylindrical grooves in the polyethylene shell on the retired tee were deformed near 9 o'clock as indicated in figure 9. The upper portions of the grooves were deformed upward/outward.

3) The longitudinal fracture through the polyethylene shell of the retired tee extended further toward the base of the tee on the inner diameter surface compared to that visible on the outer diameter surface (not shown; see optical fractography below).

4) A crack was observed in the main Aldyl A gas pipe underneath the saddle of the 2021 replacement tee as shown in figure 10. As described below, the crack was near the midline of the tee (centered approximately 0.085 inch upstream of the midline), underneath the tee's outlet, just outside the resistive fusing wires, and approximately 90° from top dead center.

2.3 Examination of retired tee for 17 S. 2nd Ave

The retired tee was visually examined and then cut into pieces in order to examine the insert and the longitudinal fracture through the tower shell. The longitudinal grooves in the shell were positioned at 1:00, 4:00, 7:00, and 10:00. As shown in figures 11a and 11b, the upper groove ends at the 10 o'clock and 7 o'clock positions were deformed upward and the longitudinal fracture in the shell was centered within the groove at 10 o'clock.

The retired tee was disassembled using a hacksaw, as shown in figures 12a – d, to expose the longitudinal fracture and the polyoxymethylene insert. First the outlet of the tee was sectioned and then the tower was cut from the saddle. The cut that removed the tower was placed low enough to ensure the insert was not also cut. The tower was then cut in the longitudinal direction at two locations, separating it into three pieces. The two cuts were positioned 135° clockwise and counterclockwise from the longitudinal fracture respectively, each cut nominally between two longitudinal grooves. The cuts were made with a hacksaw until the insert was approached and then completed with a sharp blade.

One side of the fracture (right side in figure 12d) was cleaned, first by soaking in an alkaline detergent solution and rinsing under running water, followed by a brief immersion in a sonicating bath followed by additional rinsing. The fracture surface is shown in the upper and lower images in figure 13.

The fracture surface exhibited features consistent with initiation due to slow crack growth. The upper portion of the fracture (left side in figure 13) was flat and comparatively featureless, with fibrils observed across the region, consistent with slow crack growth. This region extended through the shell wall near the top of the tower, approximately 0.233 inch across, and extended further down the tower along the inner diameter (ID) surface than along the outer diameter (OD) surface. Along the ID surface, the slow crack growth region extended approximately 1.320 inch from the top of the tower and along the OD surface, it extended approximately 0.715 inch from the top of the tower. When viewed under a stereomicroscope and oblique lighting, fine radiating crack growth features were observed on the fracture, progressing away from the inner diameter surface (see blue arrows in the lower image in figure 13). The features were traced back toward a region, indicated in figure 13, extending from 0.325 inch to 0.430 inch from the top of the tower. As shown in figure 14 (Note: photo taken after the group exam), the crack origin occurred along an impression in the shell wall created by the mold line on the

polyoxymethylene insert. Further toward the base of the tower, the fracture surface exhibited flat featureless regions transitioning to hackle consistent with a transition to fast fracture. In total, the longitudinal fracture extended approximately 2.300 inch from the top of the tower on the ID and 1.860 inch on the OD. Other than the mold line impression, there were no other apparent anomalies in the vicinity of the crack origin.

After the group activity, but included in this section for completeness, additional cracks were observed in the tower shell, as shown in figures 15 and 16. Figure 15 shows the longitudinal groove in the shell at 7 o'clock. Two curved cracks were observed in the groove near the top of the shell between the top and third from the top circumferential grooves. An incipient crack was also found near the origin of the longitudinal crack, shown in figures 16a and b.

The tower insert was fractured in the transverse plane above the lowest circumferential rib. The height of the remaining piece was approximately 0.720 inch. The insert from the sister tee measured 2.318 inch by micrometer, resulting in an approximate remaining length fraction for the fractured insert of 0.31. An image of the transverse fracture is shown in figure 17 and side views at 90° rotation intervals are shown in figures 18a – d. The insert was not cleaned and some regions of the fracture surface were covered by dirt/debris. Two ratchet marks were observed originating from the ID surface, near the 10 o'clock and 11 o'clock positions, and from 6 o'clock to 12 o'clock by way of 9 o'clock, the inner half of the through-wall fracture exhibited a crazed and fibrous appearance. By contrast, the outer half, where visible, exhibited a granular and porous appearance. From 6 o'clock to 12 o'clock by way of 3 o'clock, the entire through-wall fracture exhibited a granular and porous appearance. The fracture intersected and followed a thread root on the ID surface, progressing along clockwise and counterclockwise paths until meeting at a point of final fracture between 2 o'clock and 3 o'clock, where the two paths intersected, by jumping across a thread crest. The features were consistent with fracture initiation on the 9 o'clock side of the insert.

The insert exhibited features consistent with material degradation and volume loss. As seen in figures 17 and 18, portions of the fracture surface exhibited a granular porous appearance. Mud cracking features were observed adjacent to the fracture in some regions, as shown between 9 o'clock and 6 o'clock in figure 18a. Multiple cracks and porosity were observed in the circumferential rib and additional cracks were observed on the barrel's outer diameter surface. As shown in figure 19, the thread profile was diminished on a region of the ID surface between 6 o'clock and 9 o'clock. At a later date (not part of the group activity) the insert was sectioned for external lab testing. As part of sample acquisition, longitudinal breaks were made at 6 o'clock and 4 o'clock. The broken faces, as shown for the 6 o'clock face in figure 20, revealed that the outer portion of the insert body exhibited a granular and friable appearance. By contrast, the inner half of the wall exhibited a comparatively smooth fracture typical of fully consolidated material.

2.4 Additional flow rate testing

The outlet on the retired tee, previously cut from the tee body, was joined to a piece of 1-1/4" NPS polyethylene pipe with a coupling and leak tested as described above (see figure 12b). The outlet, service line, an installed AMP fitting, and end cap were pressurized to 54 psig. There was no measurable flow within the detection limit of the meter (0.03 SCFH). The assembly was capped and left to hold pressure for 30 min, resulting in a drop between 0.0 psig and 0.5 psig over the 30 min interval.

2.5 Steam pipe segment

A segment of the steam pipe, approximately 46 inch in length, is shown in figures 21 and 22. The pipe had a nominal outer diameter of 4.00 inch and a wall thickness between 0.20 inch and 0.22 inch, consistent with NPS 3-1/2-inch Schedule 40 pipe. The pipe was located approximately 23.4 inch to the west and 15.5 inch above the retired tee (center of pipe to top of tee). The steam pipe laid perpendicular to the gas main, with steam flowing from 77 S. 2nd Ave to 17 S. 2nd Ave (from south to north). In the middle of the segment, the pipe was corroded around its outer diameter surface. Little to no corrosion was apparent on the inner diameter surface. In the corroded region, the pipe was deformed and cracked. Viewed from the side, the pipe lengths upstream and downstream of the corroded region exhibited a vertical shear displacement relative to one another. The steam pipe segment was marked with the approximate location of the Aldyl A gas main centerline near its downstream end. See section 3.4 for distance measurements of various features along the length of the pipe.

The pipe was cracked on its east side. Assigning clock positions to the pipe, viewed from the south looking north and with top dead center as 12 o'clock. The center of the cracked region was located at 3 o'clock. The east side was deformed inward downstream of the sheared and cracked region. The cracks in the pipe ran at inclined angles to the longitudinal axis of the pipe. One crack was associated with an outward compressive bulge/buckle (figure 22b) that measured approximately 4.05 inch in length. Two transverse cracks branched off this crack, one measuring approximately 1.14 inch (toward the pipe top) and the other measuring 1.26 inch (toward the pipe bottom), respectively. The fracture faces associated with the buckle overlapped. In total, the features were consistent with initial localized shear deformation of the pipe in the corroded region followed by cracking of the pipe wall and additional torsion deformation.

2.6 Marker balls

The in-service marker ball and an exemplar marker ball are shown in figure 4b. Additional images of the in-service marker ball are shown in figures 23a and b. Marker balls are hollow spheres that contain a fluid and a passive antenna. The spheres are assembled from two polymer half-domes that are welded together. The

in-service marker ball was recovered from the excavation site for the 17 S. 2nd Ave active and retired service tees. It was found south of and above the retired tee. See the Operations Group Chairman Factual Report for additional details. As seen in figures 23a and b, both half-domes of the in-service marker ball had collapsed inward, with one dome collapsing inward more than the other. The seam joining the half domes is shown for the in-service and exemplar marker balls in figures 24a and b, respectively. For the in-service ball, a gap was observed at the seam between the two half-domes. By contrast, for the exemplar ball, the seam was sealed by a plastic bead.

The in-service marker ball had lost much of its levelling fluid. According to the manufacturer's product literature, the nominal weight of a ball is 350 g. The exemplar ball weighed 364 g while the in-service ball weighed 175 g. Some small amount of levelling fluid remained inside the in-service ball as it could be seen and heard inside the ball when it was shaken.

3.0 Follow on examinations

After the completion of the group exam, additional laboratory work was conducted on the 77 S. 2nd Ave tee (sister tee), 17 S. 2nd Ave active service tee (2021 replacement tee), the cutter from the retired tee, the recovered O-ring, sister tee O-ring, and the steam line. An external laboratory was also contracted to perform additional testing on the 17 S. 2nd Ave retired service tee (retired tee).

3.1 Sister tee follow-on examination

An effort was made to locate the small leak in the sister tee assembly, noted during the group exam, but it could not be located. The pipe ends were plugged (with a different set of internal pipe plugs than those used during the group exam), the assembly was pressurized to 35 psig, and the assembly was submerged in a water tank. Bubbles were observed leaking past the plugs, but no bubbles were observed originating from the tee or elsewhere along the pressurized segments of pipe.

The cap on the service tee was removed by hand, requiring significant force to free it. There was a yellow-colored grease-like substance on the threads of the cap, threads of the insert, around the edge of the cutter head, and the top face of the insert, as shown in figure 25. A sample of the substance was examined by a Fourier-transform infrared spectrometer (FTIR) equipped with a diamond crystal attenuated total-internal reflectance (ATR) bench accessory and the spectrum was matched using the "KnowItAll" online spectrum library (Wiley Science Solutions). The best match was for a "Heavy White, Moisture-proof grease". The grease was removed from the fitting with a scalpel and set aside in a glass sample vial.

The pipe ends were plugged again, and the tee was pressurized to 30 psig without the tower cap installed. The observed flow rate was 0.27 SCFH, consistent with gas flowing past the cutter threads.

The cutter was turned into the hole and then retracted from the hole while the torque was measured by a torque transducer and the tee was under 40 psig pressure. Peak torque for the sister tee was approximately 32 lbs-in on insertion. The cutter was fully extracted from the tower and set aside after the test.

Visual examination of the tower shell, insert, and cap did not reveal any notable features. No dirt or debris was observed in the tower and there was no apparent damage to the insert or cap. The insert was exposed using a similar hacksaw cutting method as used for the retired tee (refer to figure 8a). Comparing the color of the sister tee's and retired tee's polyethylene shells, the retired tee exhibited a bleached appearance, particularly on the inner diameter surface. The insert had a die number embossed on it, visible parting lines on opposite longitudinal ribs, and ejector pin outlines. Imprints of these features were observed on the inner diameter surface of the polyethylene tower shell.

FTIR fingerprinting using the ATR accessory confirmed the following materials used in the tee: tower shell – polyethylene; insert – polyoxymethylene (polyacetal); cap – also polyoxymethylene; cutter head – polyamide (nylon). See figures 26a – d, respectively.

3.2 2021 replacement tee follow-on examination

The crack in the 1-1/4" Aldyl A pipe adjacent to the 2021 replacement tee was examined and the fracture surfaces exposed. The undersaddle, used to hold the saddle against the pipe during the fusing/welding process, was removed. A section on the underside of the Aldyl A pipe was cut to visually examine the area where the crack was observed in the CT scan, as shown in figures 27a and b. The crack was visible on the inner diameter surface, measured approximate 0.39 inch in length, and was centered approximately 0.085 inch upstream of the tee midline on the same side as the tee outlet. Using the X-ray CT data and visual observations, a transverse section of the saddle was removed that fully contained the crack, as shown in figures 28 and 29. The through-wall section was ground on 600-grit silicon carbide paper to evaluate the fusion zone. No apparent cracks, gaps, or voids were observed. The crack was then exposed by backcutting with a utility knife.

Examination of the fracture surface indicated that the crack initiated on the outer diameter surface of the pipe, as indicated in figure 30. Multiple arrest marks and cyclic crack growth features were observed. Undulating slow crack growth features emerged closer to ID surface. The features were consistent with progressive crack growth.

3.3 Cutter from retired tee

The cutter head from the retired tee exhibited multiple cracks and fractures and exhibited a region of surface roughening and surface profile changes. Images of

the cutter head are shown in figures 31 – 33. Multiple transverse and longitudinal cracks were observed around the barrel and a transverse crack ran across the floor of the socket. The majority, but not all, of the transverse cracks initiated at thread roots on the OD surface. One region of the cutter head exhibited a roughened appearance, as seen in figure 33. In this region, the surface profile was altered, with thread crests rounded off and smoothing of a sharp-radius barrel transition. The plug from the original piercing of the Aldyl A pipe was found deep inside the hollow metal cutter.

The chemical composition of the metal portion of the cutter was identified using a handheld X-ray fluorescence spectrometer. The spectrometer identified the metal at 304 stainless steel.

3.4 O-rings

Images of the O-ring recovered from the 17 S. 2nd Ave service tee excavation site and the sister tee providing gas service to 77 S. 2nd Ave are shown in the left and right images of figure 34, respectively. The O-rings were of similar size and profile. The thickness, width, and outer perimeter of each O-ring were measured using a micrometer and digital optical microscope as required. Thickness and width measurements were performed four times at 90° intervals. The thickness of the recovered O-ring ranged from 0.089 inch to 0.094 inch. The thickness of the sister tee O-ring ranged from 0.089 inch to 0.090 inch. The width of the recovered O-ring ranged from 0.110 inch to 0.113 inch. The width of the sister tee O-ring ranged from 0.111 inch to 0.112 inch. Finally, the perimeter of the recovered O-ring measured 4.417 inch and the perimeter of the sister tee O-ring measured 4.465 inch.

The recovered O-ring was split at an oblique angle at one location as shown in figure 35. There was a bulge on one face of the ring at this location. The ring exhibited features consistent with aging of the rubber. Figures 36 a and b show images of the ring outer diameter surface 90 degrees clockwise and counterclockwise from the split, respectively. The O-ring was blistered in both locations, but the blisters were more prevalent for the location shown in part a. Qualitatively, the recovered O-ring was stiffer and less flexible than the O-ring from the sister tee.

3.5 Steam pipe segment

The corrosion pattern on the steam pipe was visually assessed. Starting at the south (upstream) cut end, the OD surface appeared comparatively free of notable wall loss for the first 16 inches. The ends of the crack associated with the buckled wall started and stopped at approx. 20-1/2 inch and 24-3/8 inch relative to the upstream cut end. A point micrometer was used to measure the pipe wall thickness at the edge of the buckled crack at two places and measured 0.038 inch and 0.042 inch. For a nominal wall thickness of 0.226 inch for Schedule 40 pipe, the estimated remaining

wall thickness fractions were 0.17 and 0.19, respectively. The downstream end of the wall thinning region was difficult to determine visually but appeared to extend no further than 36 inch from upstream cut end (i.e., the last 9.5 inches of the pipe segment was comparatively devoid of apparent wall loss). The pipe was marked with the approximate centerline location of the gas main at 36-9/16 inches from the upstream cut end. The calculated distance between the center of the crack and the mark for the gas main was 14-1/8 inch.

The material composition of the steam pipe was identified using a handheld X-ray fluorescence spectrometer on a region of pipe that had been sanded to bare metal with silicon carbide paper. The spectrometer identified the pipe as carbon steel with approximately 0.5% manganese (Mn).

3.6 External laboratory testing

Additional testing on the retired tee and sister tee was conducted by an external laboratory. The following tests were performed:

1) Differential scanning calorimetry (DSC) of a sample from the sister tee insert to confirm the use of polyoxymethylene homopolymer (as opposed to copolymer) in manufacturing of the insert.

2) FTIR oxidation analysis on through-wall microtome cross sections from the retired tee, taken near the initiation region of the longitudinal fracture.

3) Gas chromatography and mass spectroscopy (GC-MS) from insert samples removed from the retired tee and the sister tee. The samples were taken near the base of the insert between 6 o'clock and 4 o'clock.

The peak melting temperature of the insert sample was consistent with a polyoxymethylene homopolymer. No apparent oxidation peaks were observed on the FTIR cross sections. Finally, there were no notable volatiles emitted from the retired tee sample, and the total amount of volatiles emitted from the retired tee sample was less than that emitted from the sister tee sample. See the attached laboratory report in Appendix C.

At a later date, a 15 g sample of medium density polyethylene from the retired tee was sent to the same laboratory for melt flow index measurements. The tests were performed in accordance with ASTM D-1238 at a test temperature of 190 °C and load of 2.16 kg. The melt flow index measured 1.156 g/10 min for all three samples and met the material requirements of 1.1 g/10 min ± 20% (± 0.22 g/10 min).

Submitted by:

Donald Kramer, Ph.D.
Senior Materials Engineer

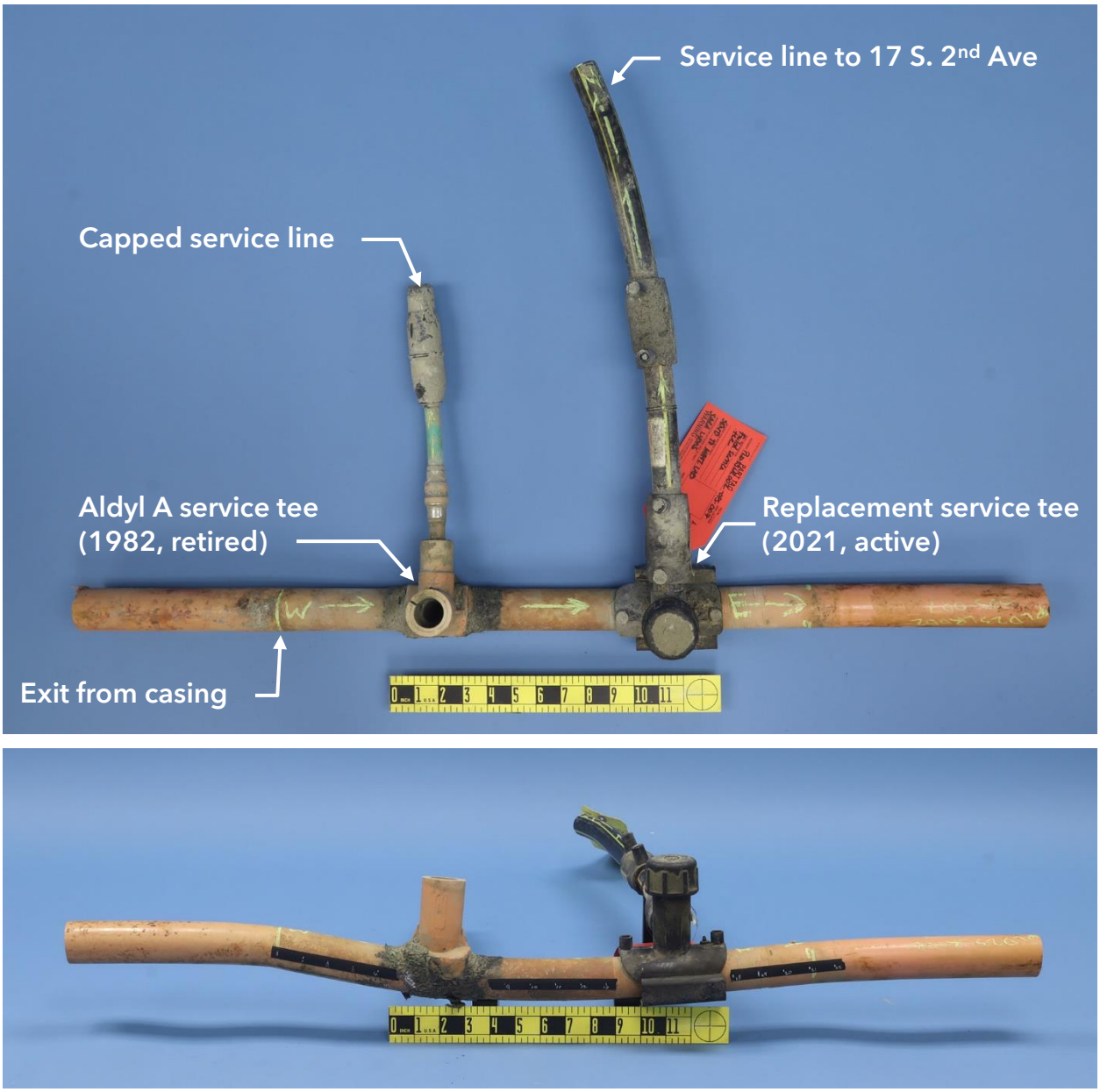


Figure 1. Aldyl A gas main with retired service tee and adjacent active service tee, installed in 2021 and providing gas service to 17 S. 2nd Ave at the time of the accident.

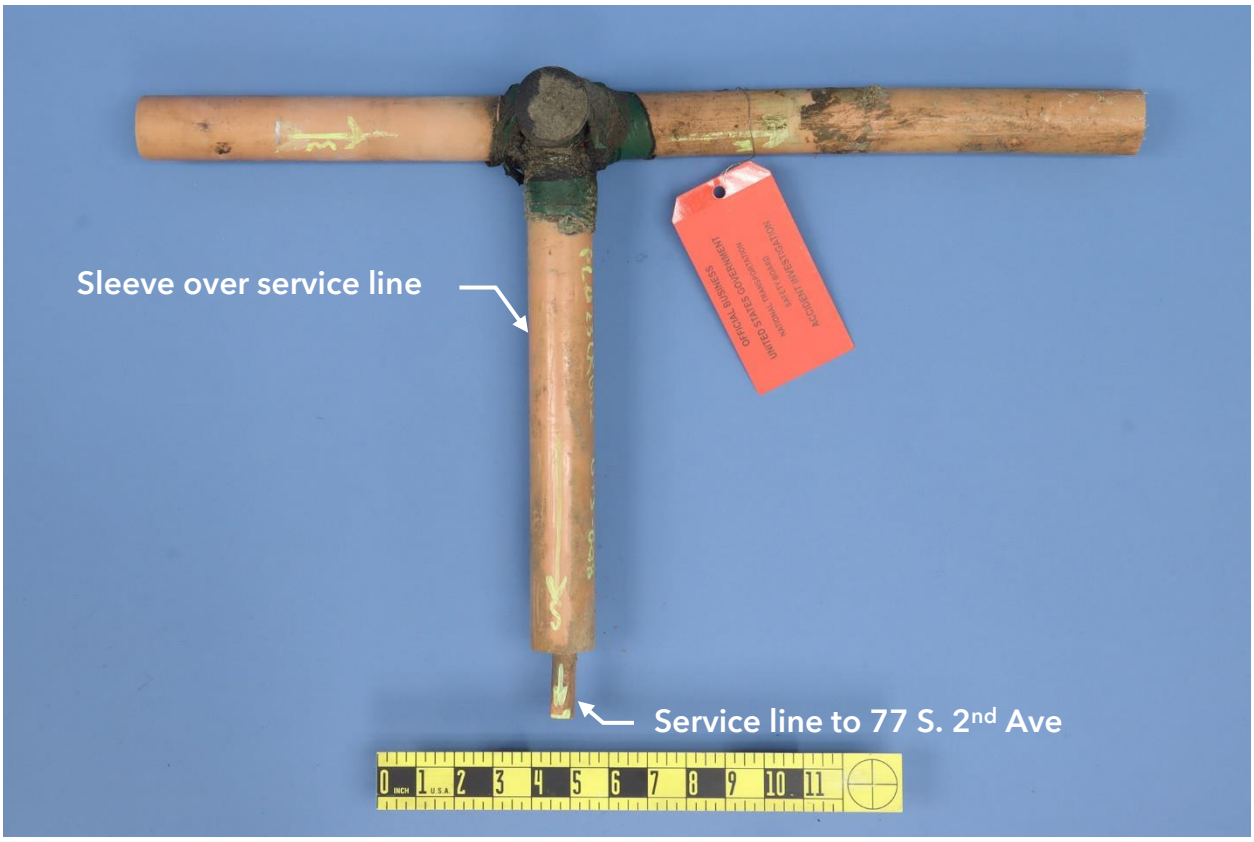
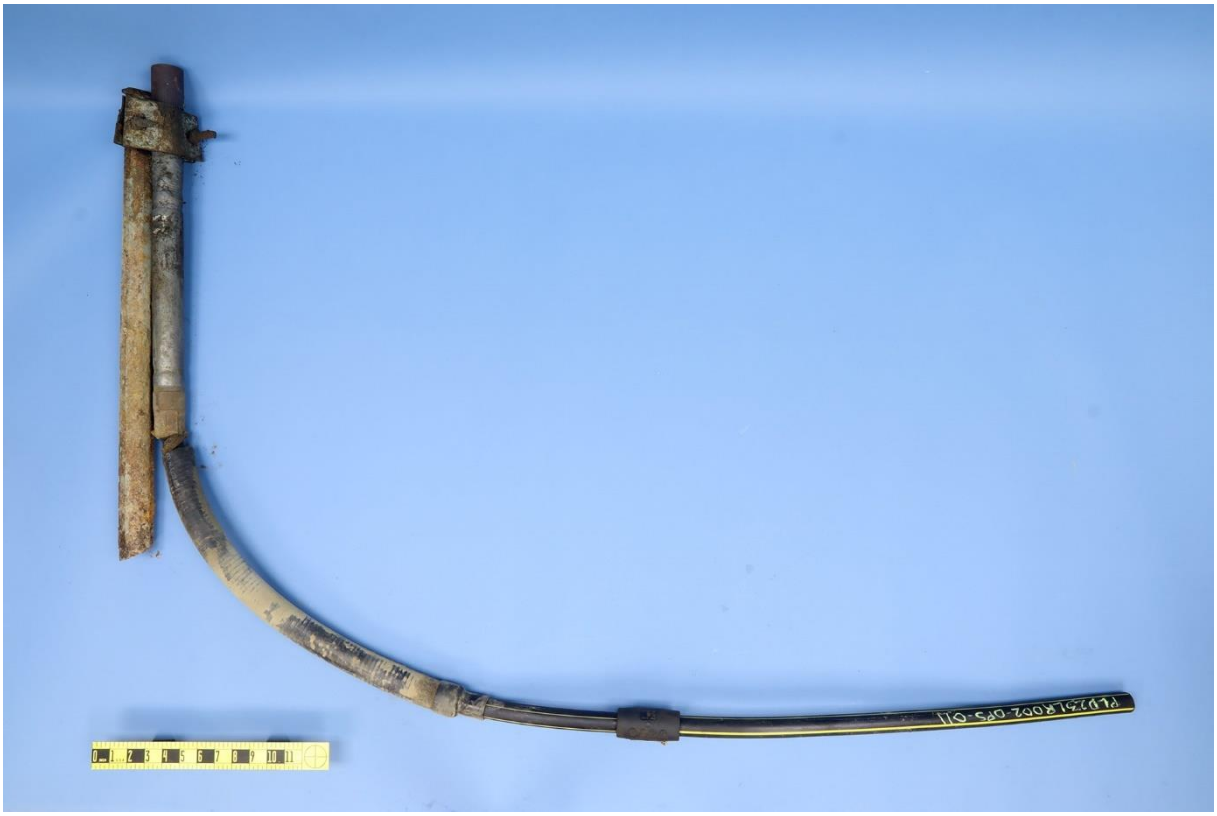


Figure 2. Aldyl A gas main segment, service tee, and service line segment for 77 S. 2nd Ave.

Figure 3. The cutter and O-ring, recovered from the excavation site for the active and retired service tees that provided gas service to 17 S. 2nd Ave (Palmer building #2).

a)



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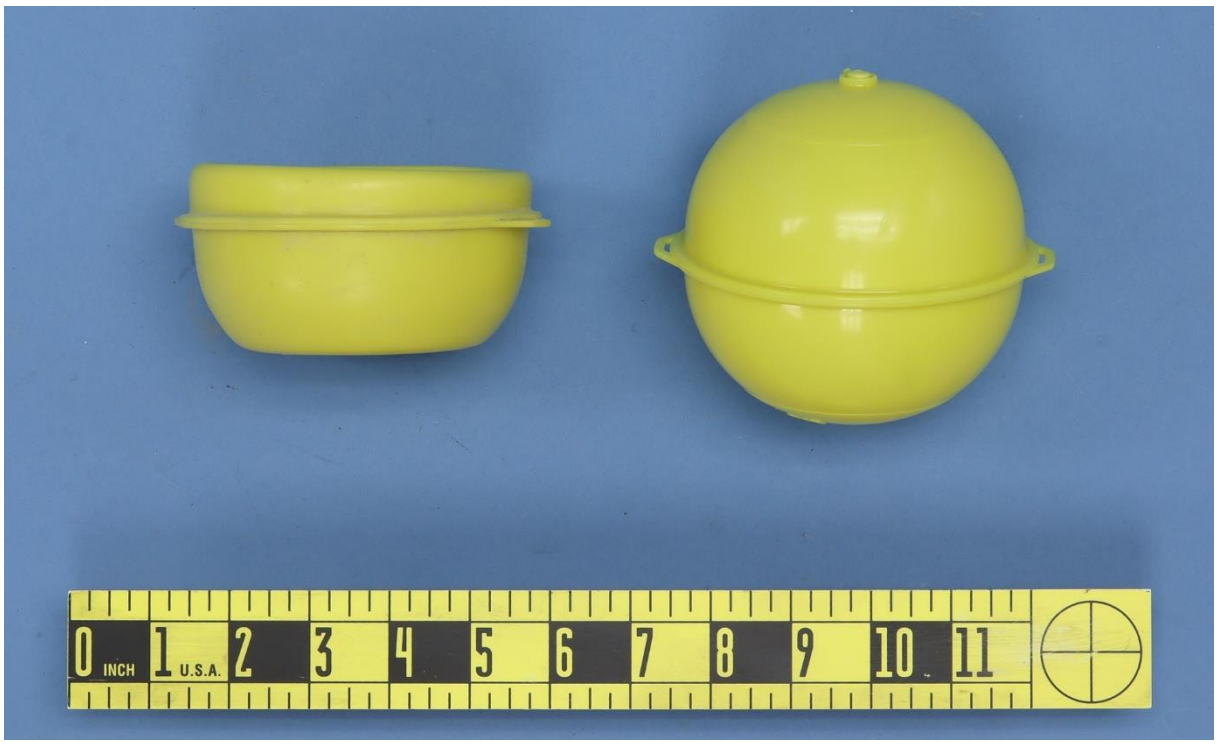
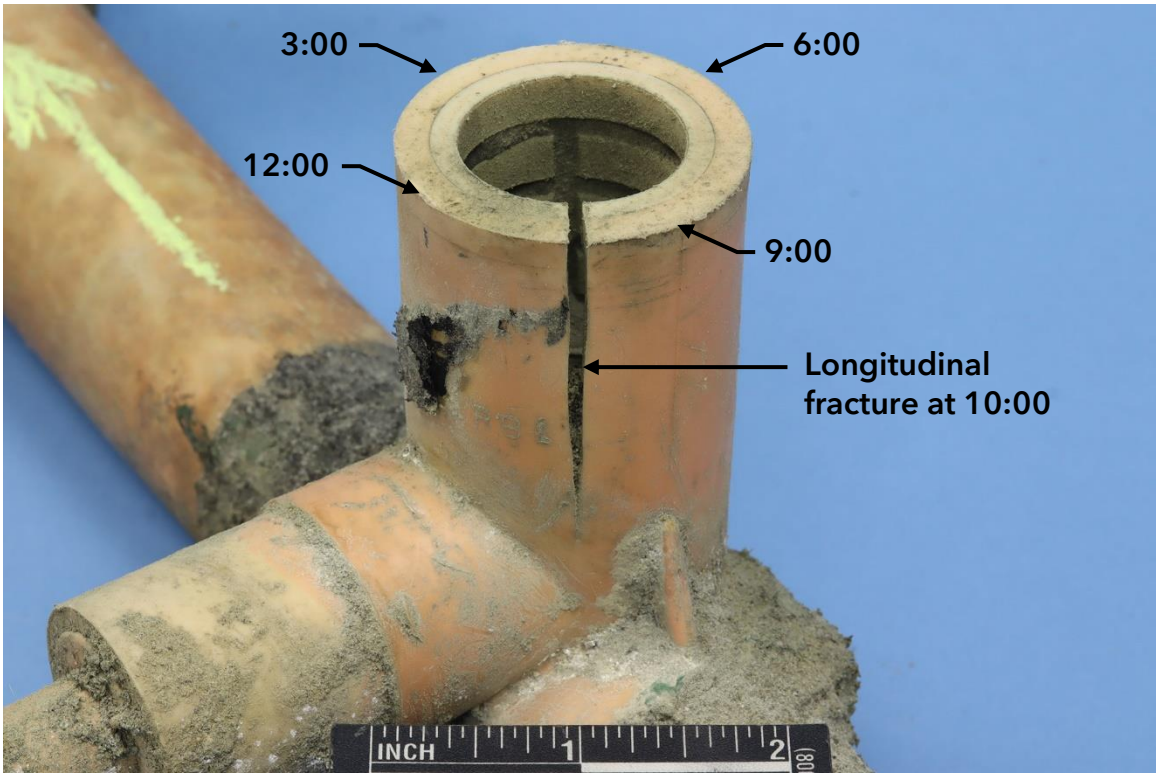


Figure 4. a) Image of 17 S. 2nd Ave gas service line and riser leading to the gas meter and b) image of marker balls. Left: collapsed marker ball located near the 17 S. 2nd Ave service tees. Right: Exemplar marker ball.

Figure 5. East side of buried steam pipe segment.

a)



b)

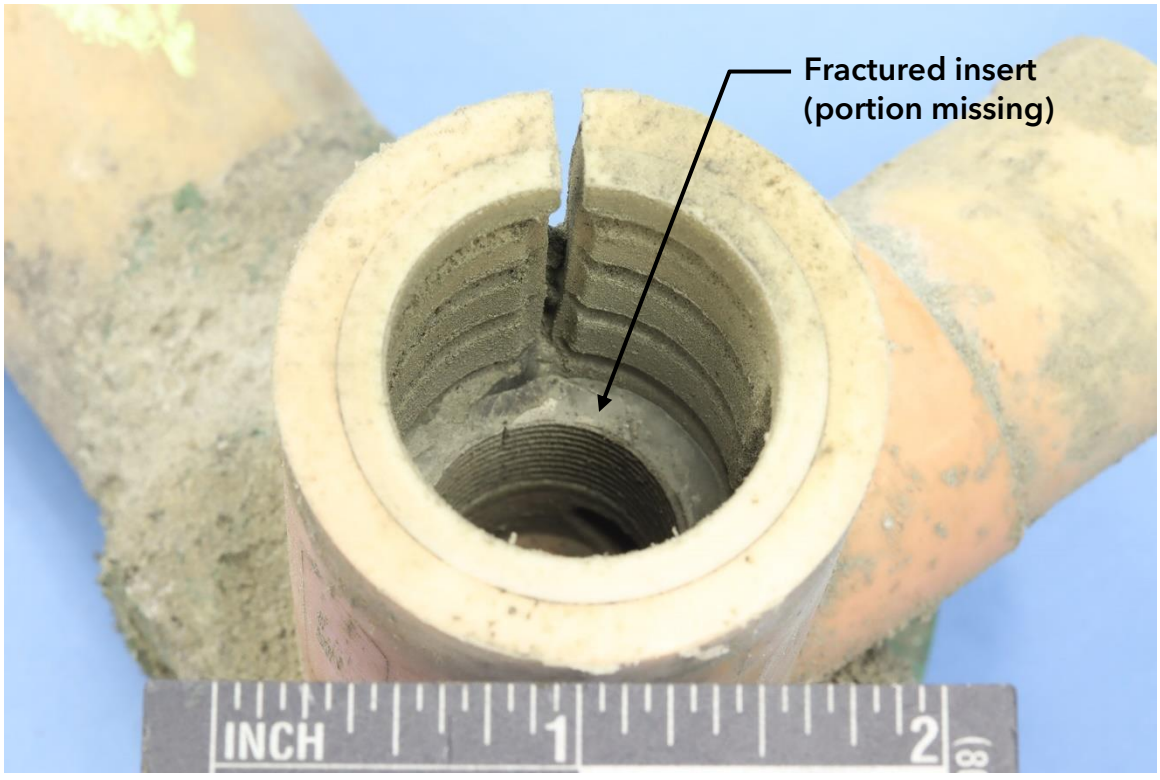


Figure 6. Images of the retired service tee from 17 S. 2nd Ave: a) image showing clock position nomenclature and the longitudinal fracture in the shell at 10:00; b) image inside the tower showing the fractured insert;

c)

d)

Figure 6 (cont.). c) image showing the appearance of the green tape on the top west side of the tower; and d) image showing the appearance of the tape on the underside of the tower.

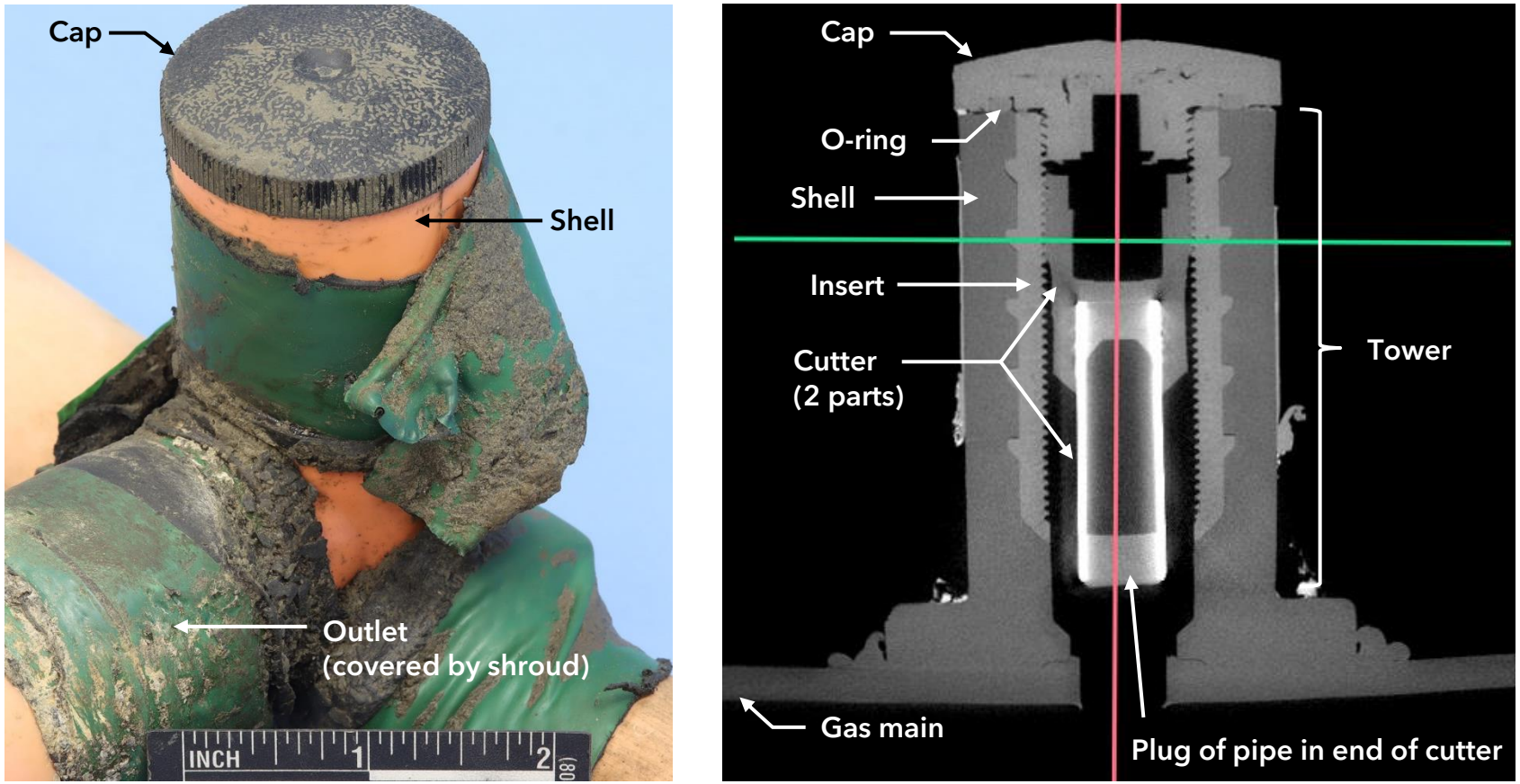
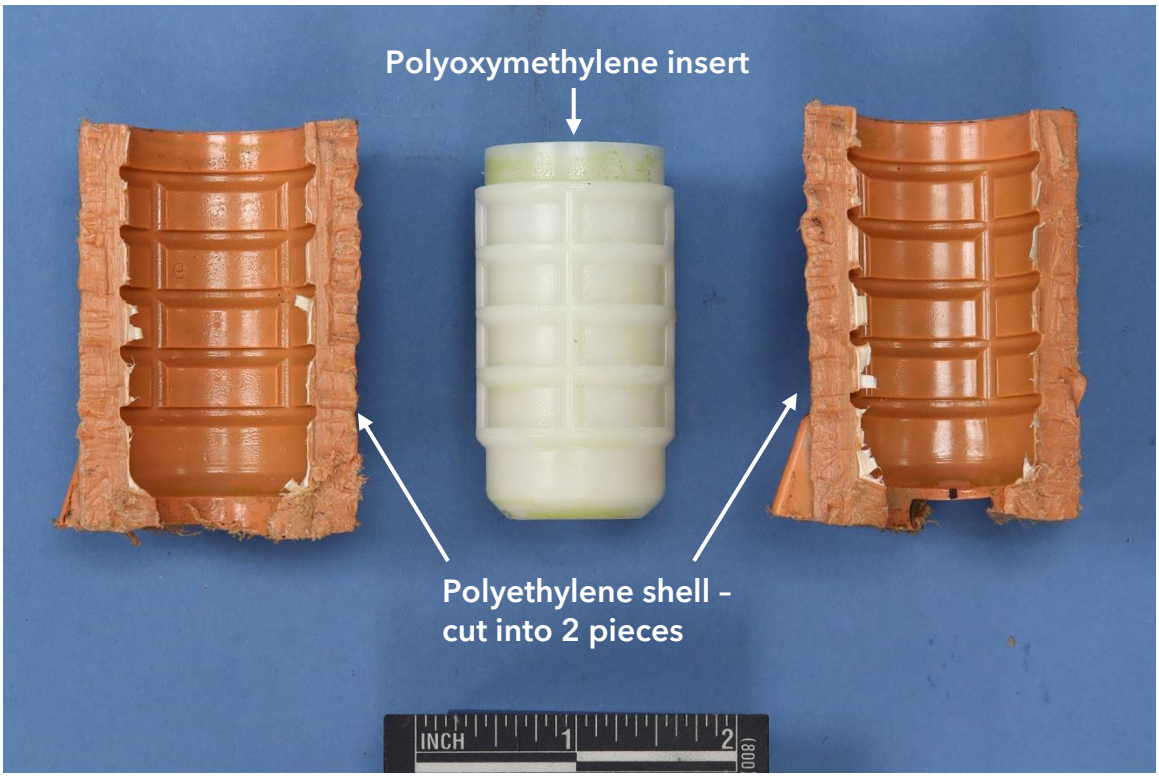


Figure 7. Images of 77 S. 2nd Ave service tee (sister tee). Left: Photograph showing external view of tower and b) X-ray CT cross section showing internal components of the tower.

a)



b)

Figure 8. Components of the service tee from 77 S. 2nd Ave: a) image of the tower components: the polyoxymethylene insert and polyethylene shell; b) image of the cutter; and

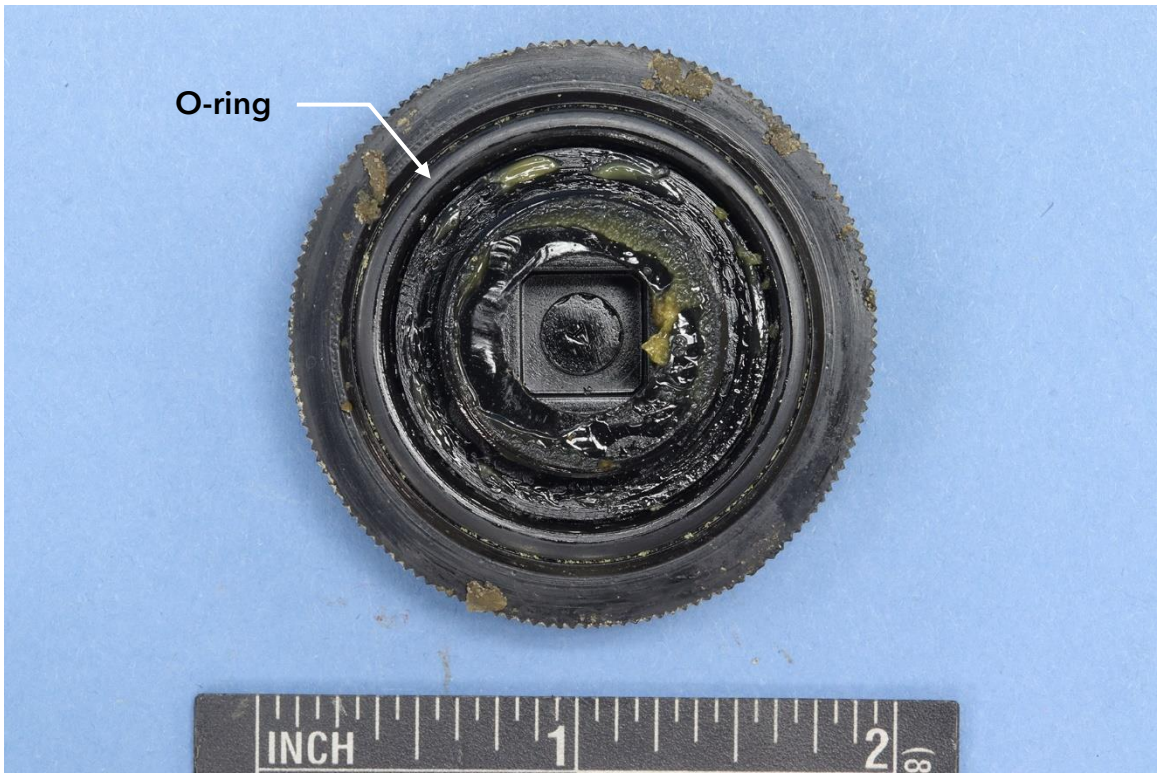
c) 

Figure 8 (cont.). c) image of cap underside with O-ring in groove.

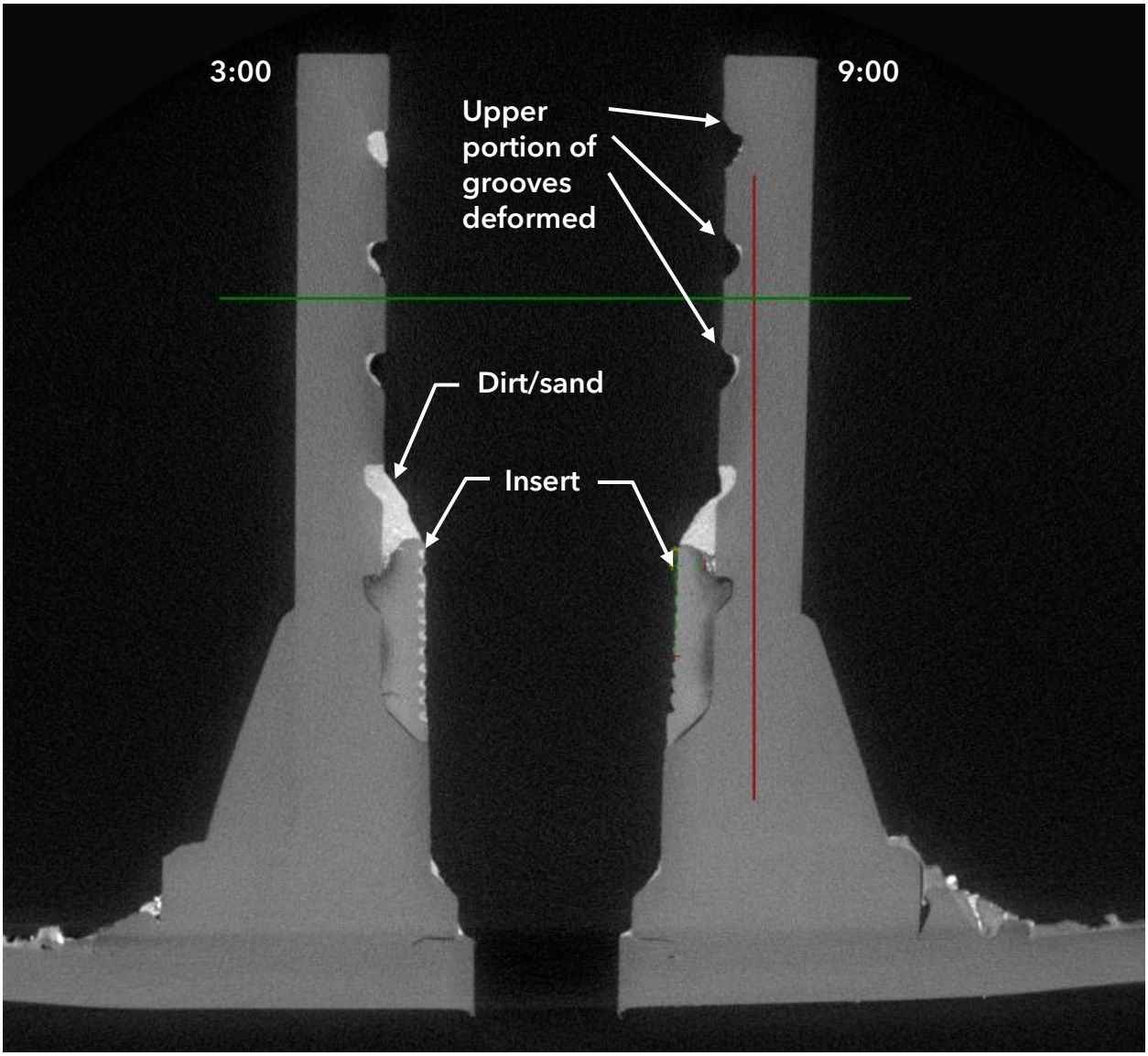


Figure 9. X-ray CT cross section of retired tee from 17 S. 2nd Ave through the 3:00 and 9:00 positions. The circumferential grooves on the 9:00 side of the shell were deformed upward/outward.

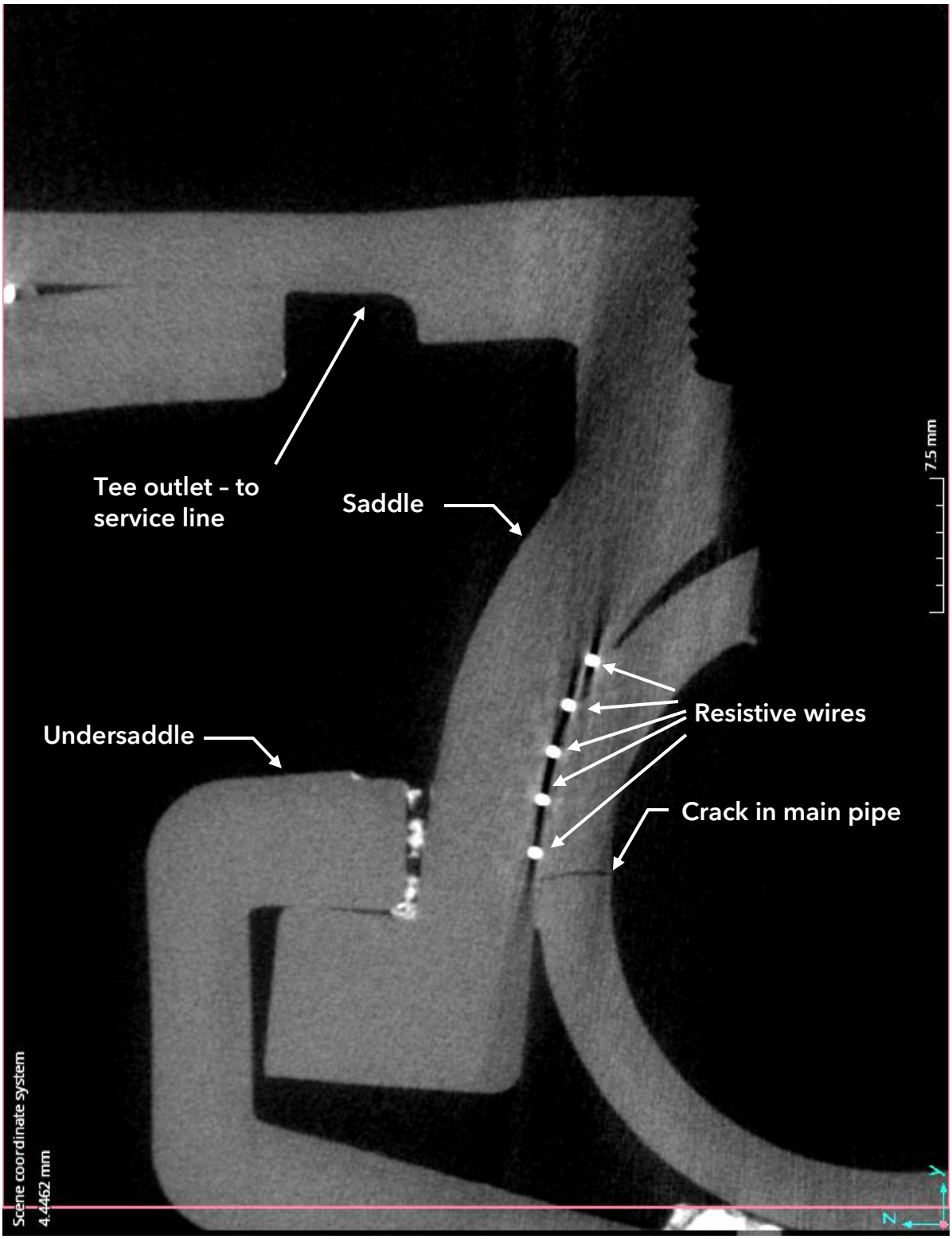


Figure 10. X-ray CT cross section through the 2021 replacement tee, viewed looking in the downstream direction. A crack was observed in the main pipe, close to the midline of the tee.

a)

b)

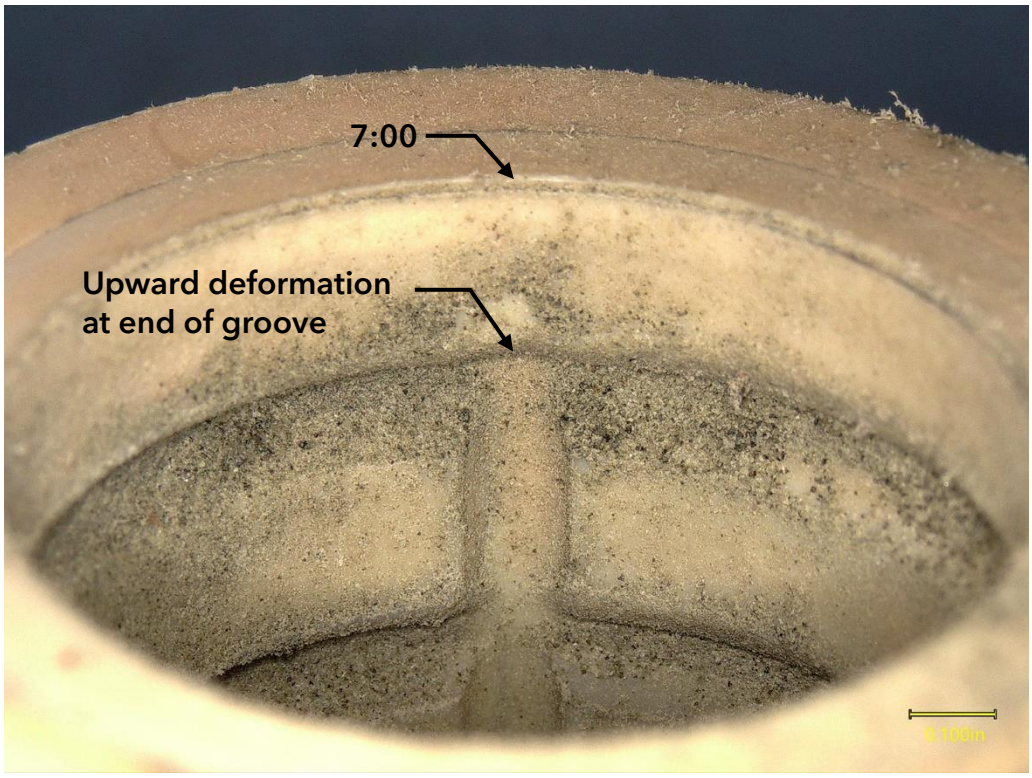


Figure 11. Images showing upward deformation at the ends of two longitudinal grooves in the retired tee polyethylene shell: a) groove at 10:00 and b) groove at 7:00.

a)

b)

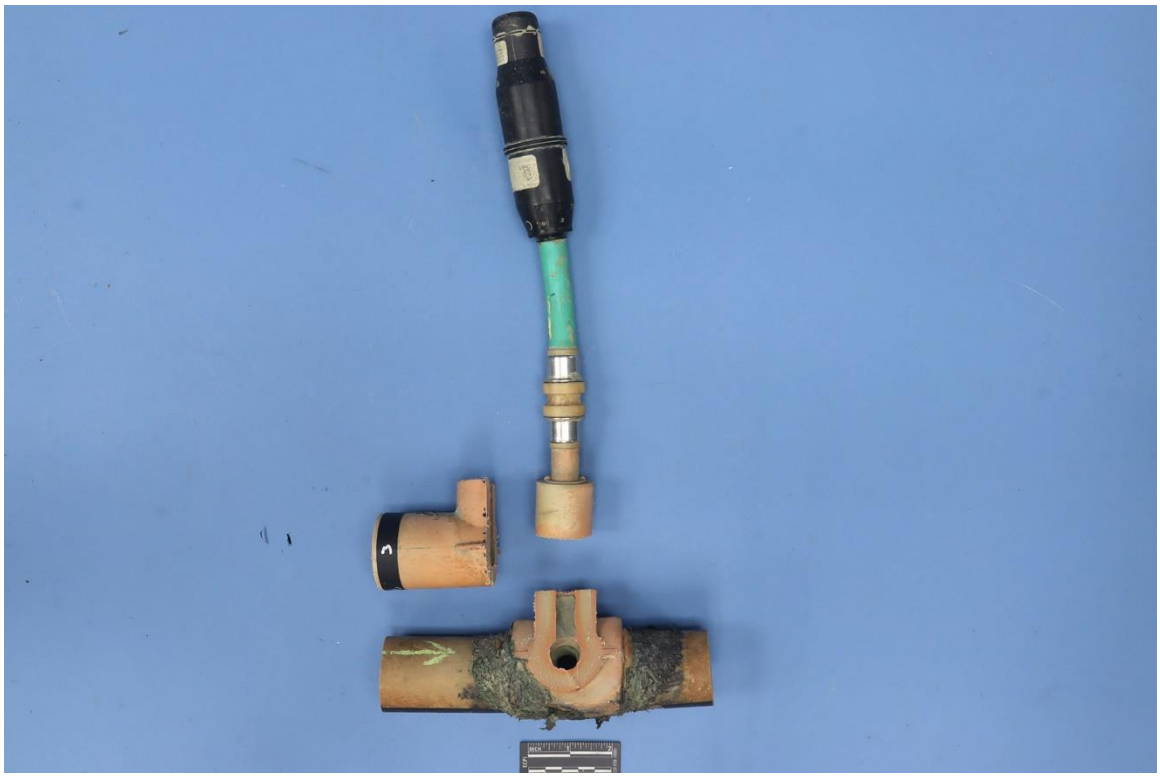
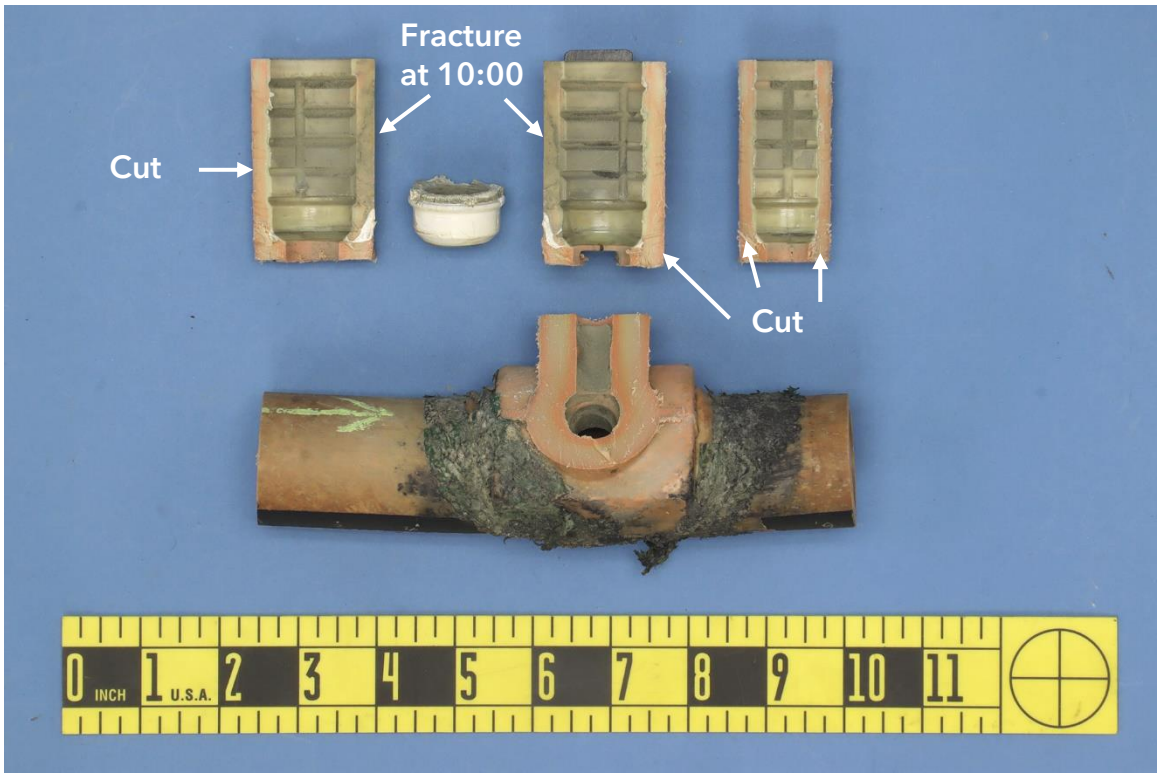


Figure 12. Images showing the disassembly of the retired tee: a) cutting of main pipe; b) cutting of service line outlet and separation of tower from base;

c)



d)

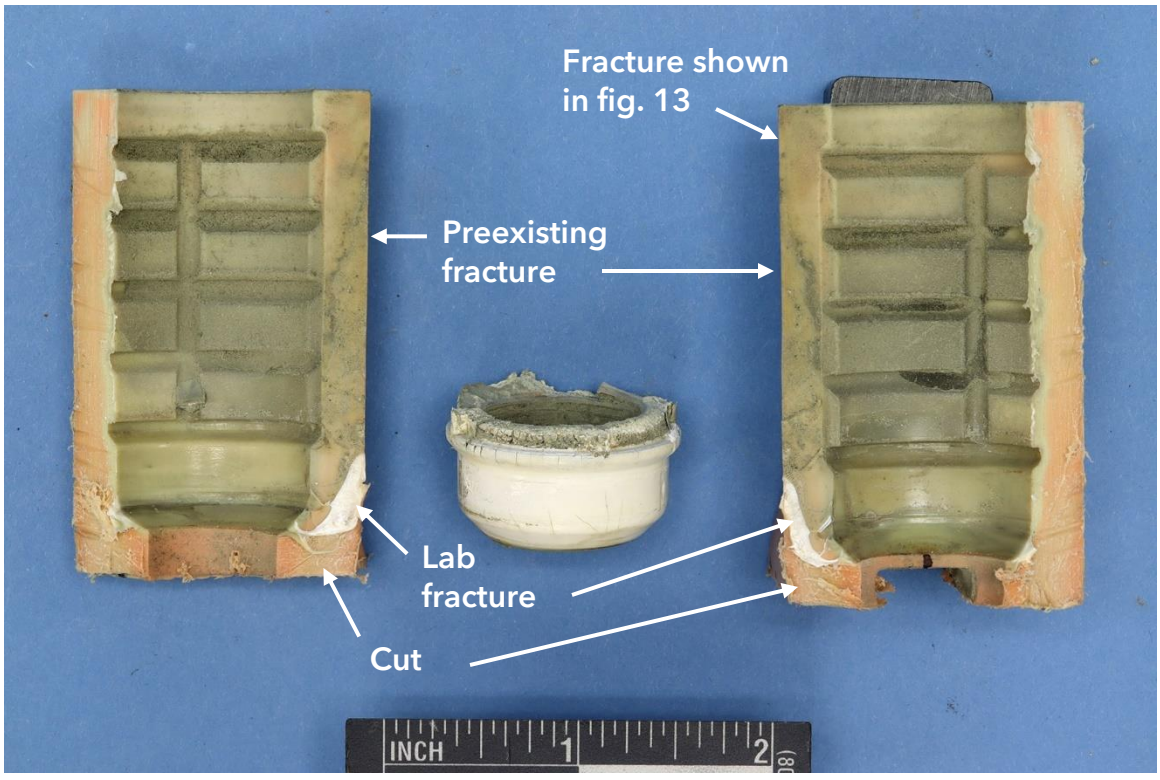


Figure 12 (cont.). c) tower sectioned into three pieces exposing the insert; and d) the two tower pieces containing mating longitudinal fracture surfaces and the insert.

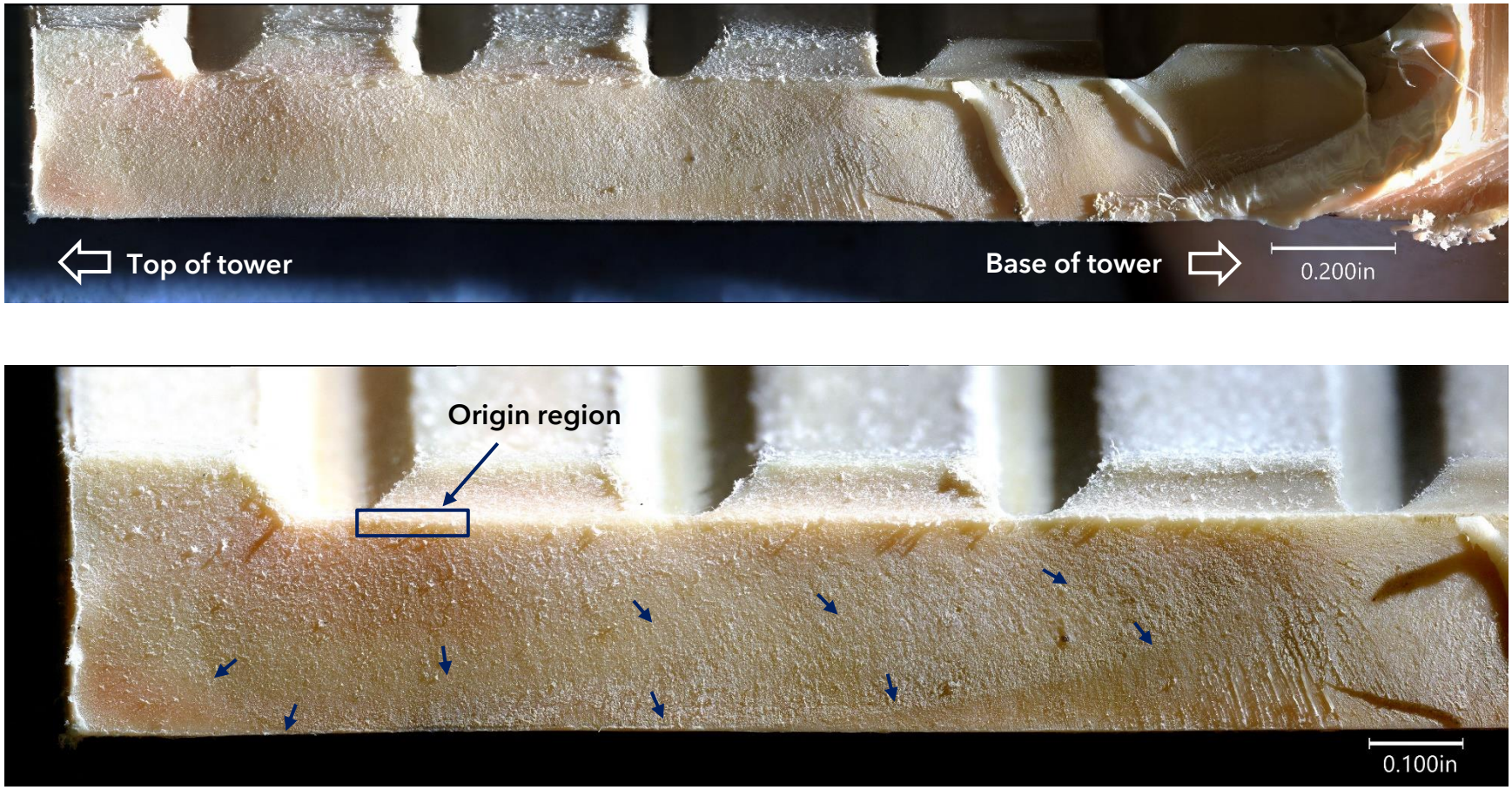


Figure 13. Upper image: Longitudinal fracture from top of tower to base of tower. Lower image: Detailed image of slow crack growth region with blue arrows indicating the local direction of crack propagation.

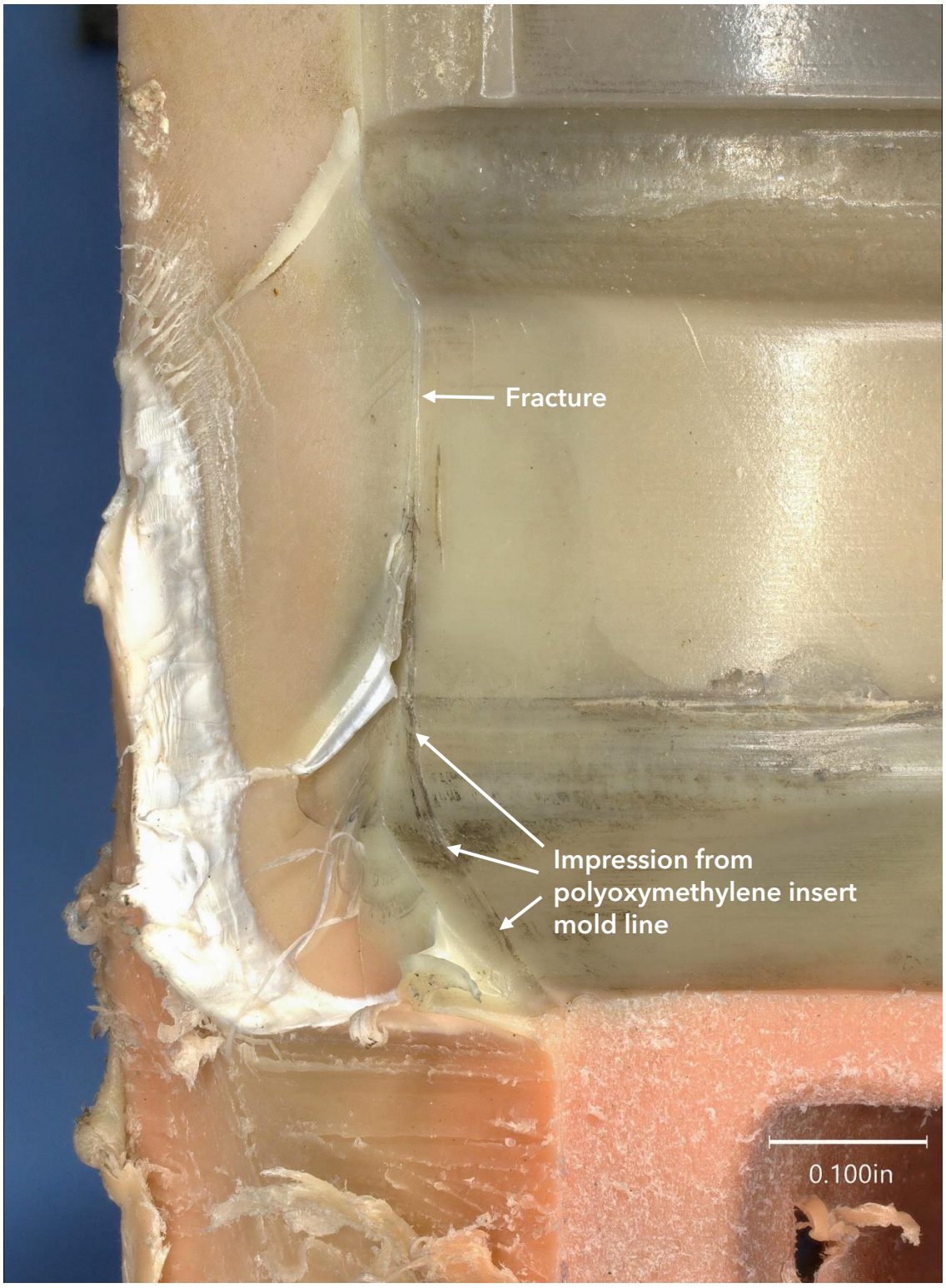


Figure 14. Image from the retired tee tower shell near the base of the tower. The longitudinal fracture coincided with an impression in the shell created by the mold line on the polyoxymethylene insert.

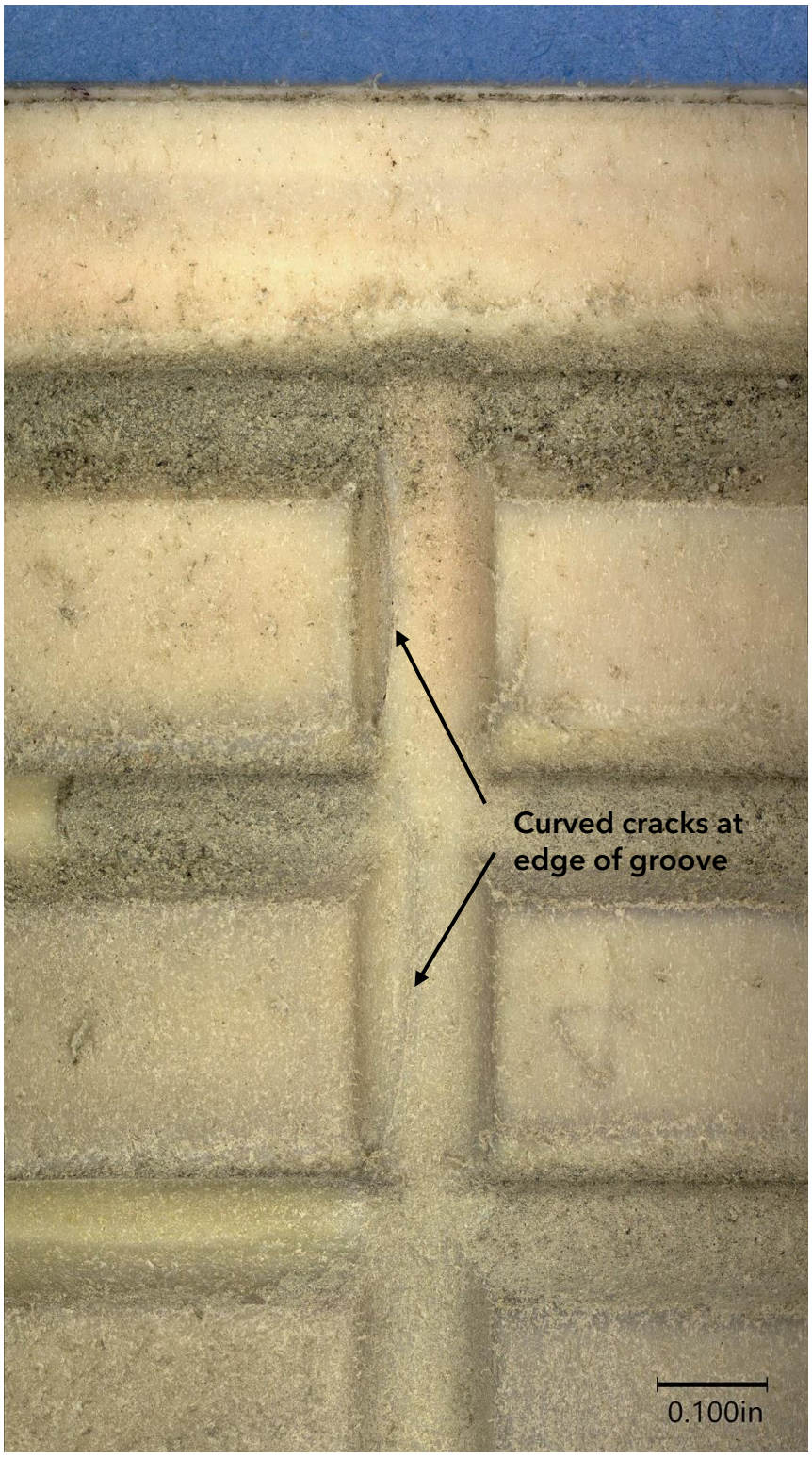


Figure 15. Image of the longitudinal groove at 7:00 in the retired tee highlighting two additional cracks.

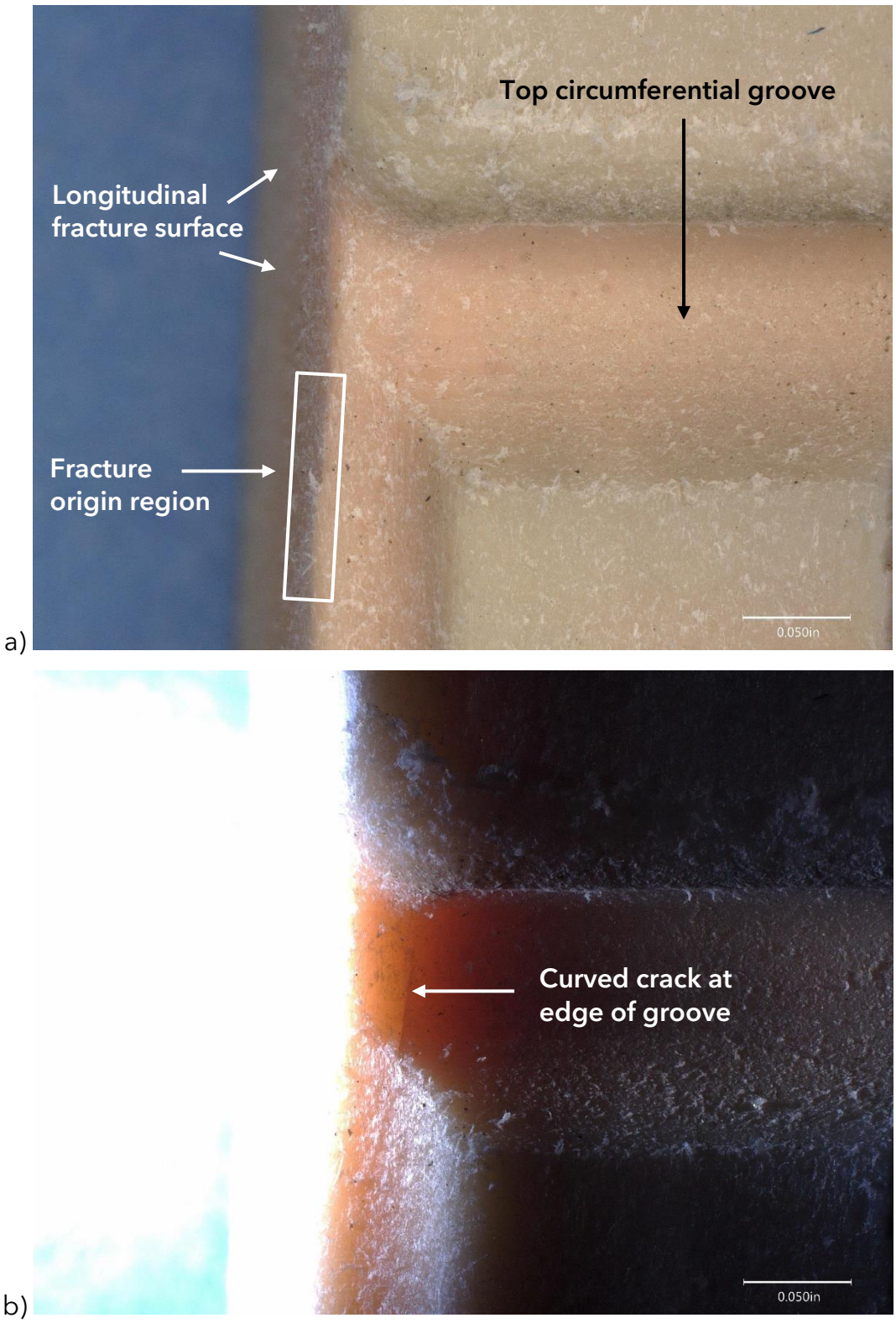


Figure 16. Shallow crack observed in the retired tee near the origin of the longitudinal fracture: a) reference image under normal lighting and b) image under penetrating oblique lighting with crack indicated.

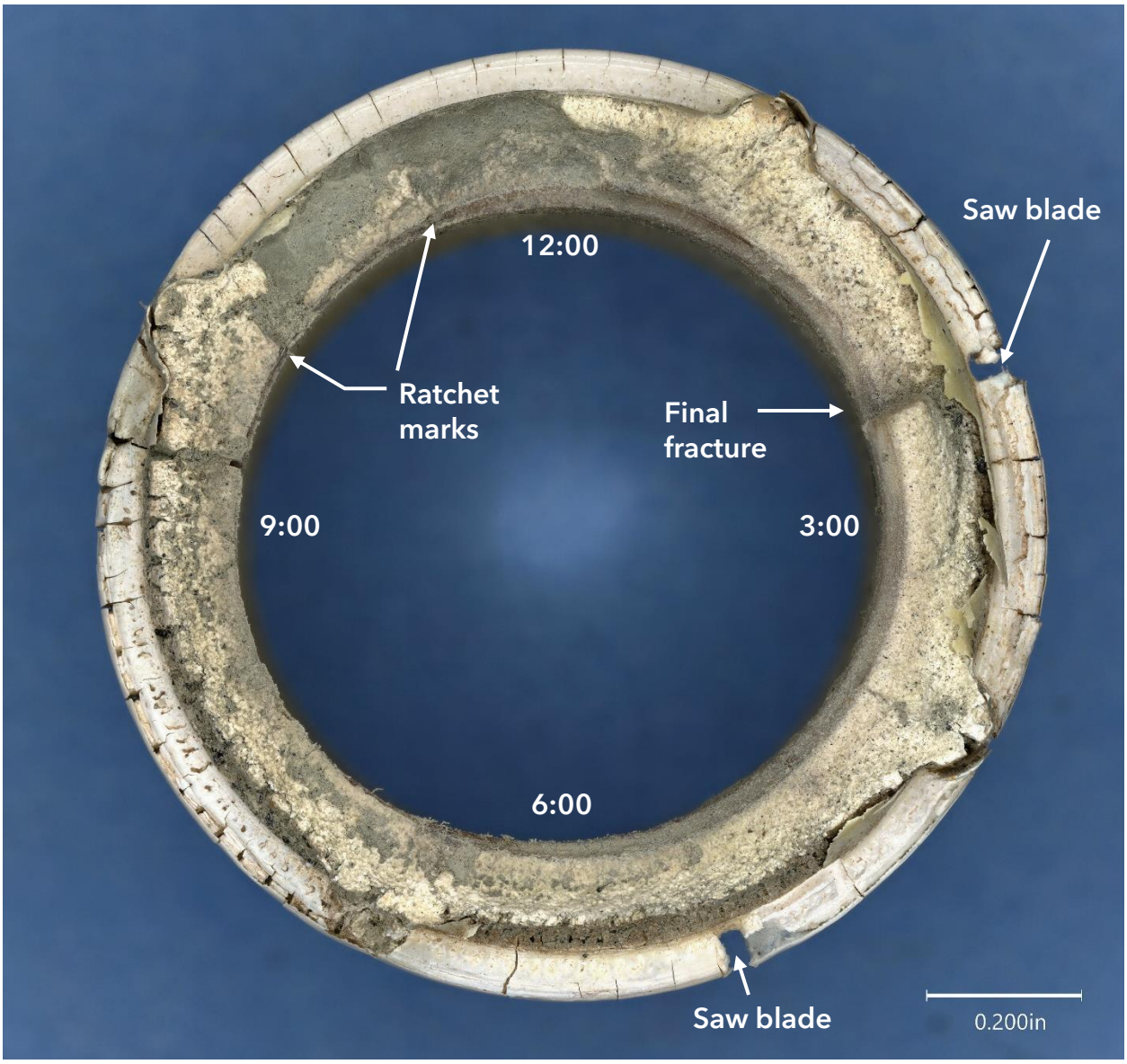
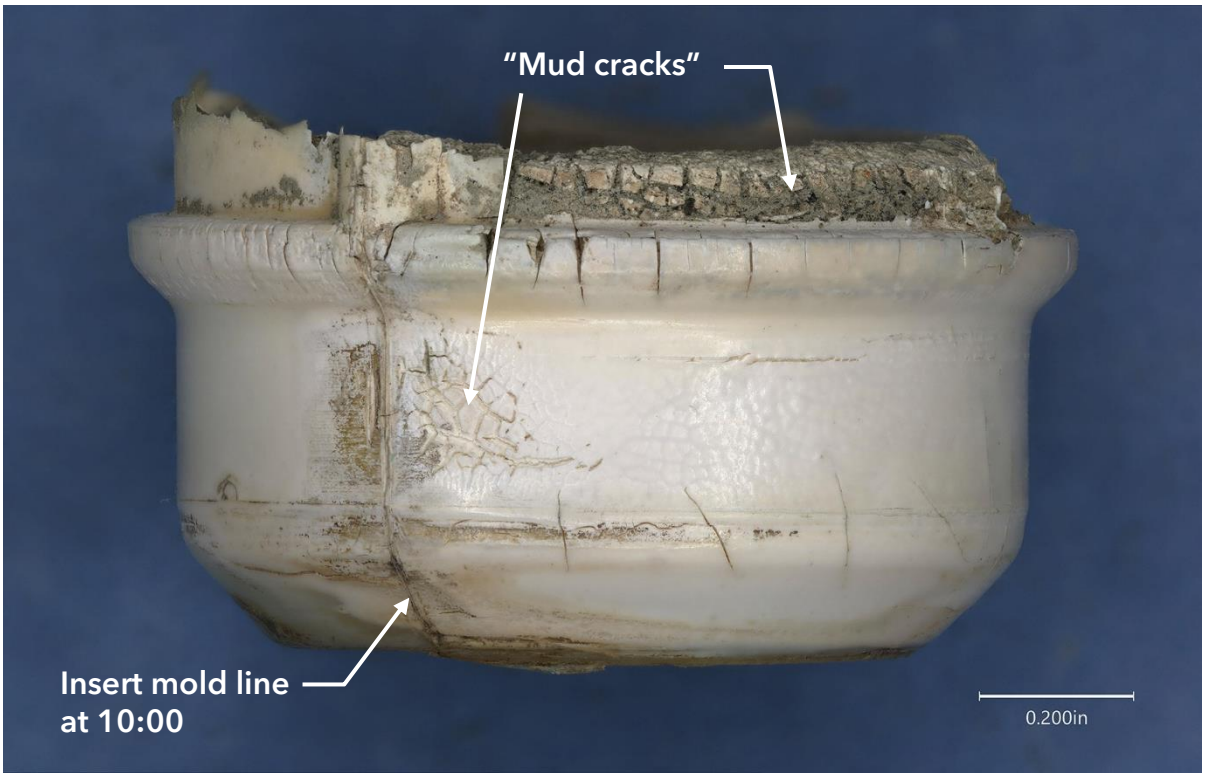


Figure 17. Image of insert fracture surface from the retired tee.

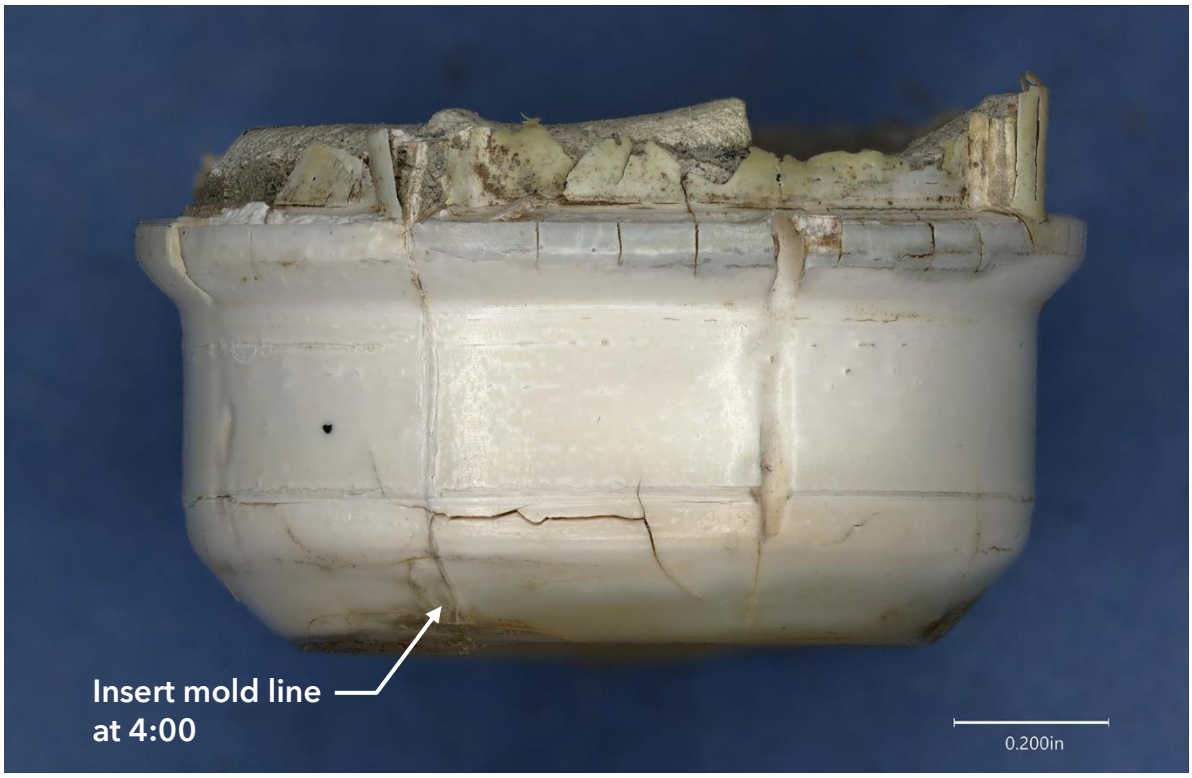
a)



b)

Figure 18. Side views of the insert from the retired tee: a) 9:00; b) 6:00;

c)



d)

Figure 18 (cont.). c) 3:00; and d) 12:00.

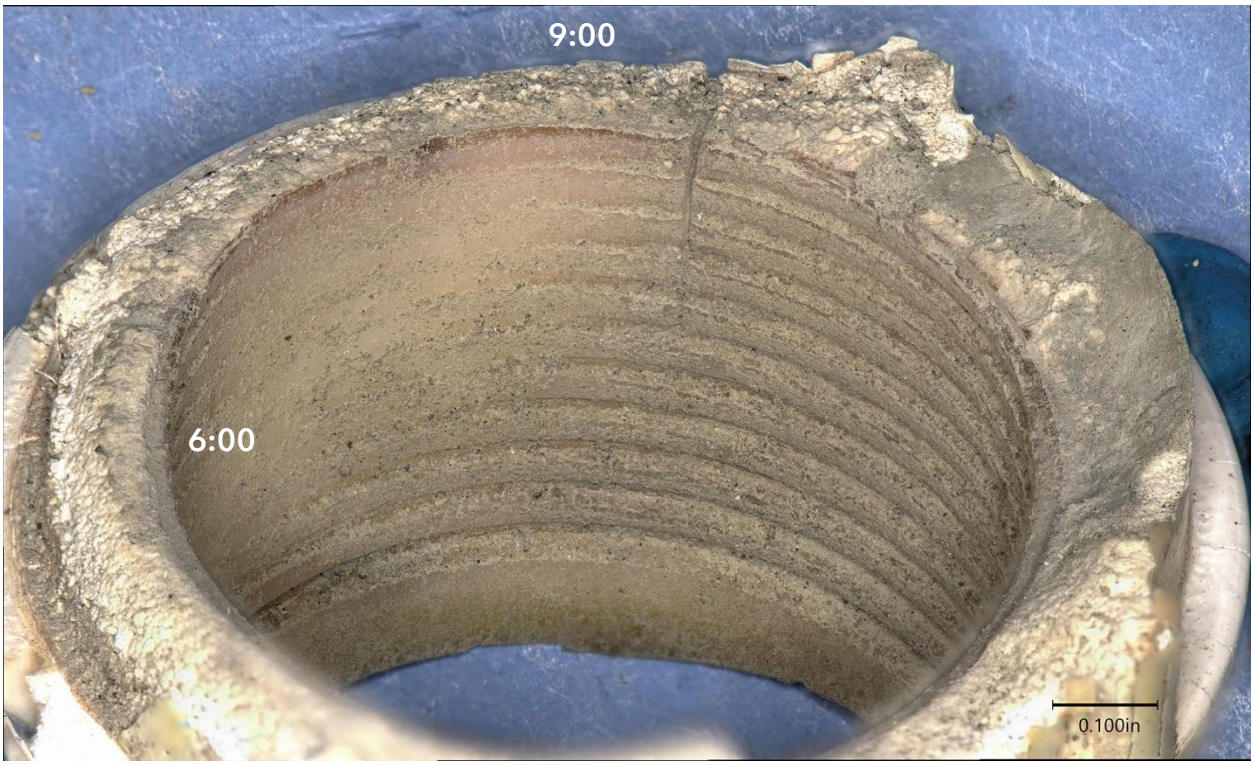


Figure 19. Image of the insert inner diameter showing loss of thread profile between 6:00 and 9:00.

Figure 20. Longitudinal cross section of retired tee insert at 6:00 showing granular friable material on the outer half of the wall and fully consolidated material on the inner half of the wall.

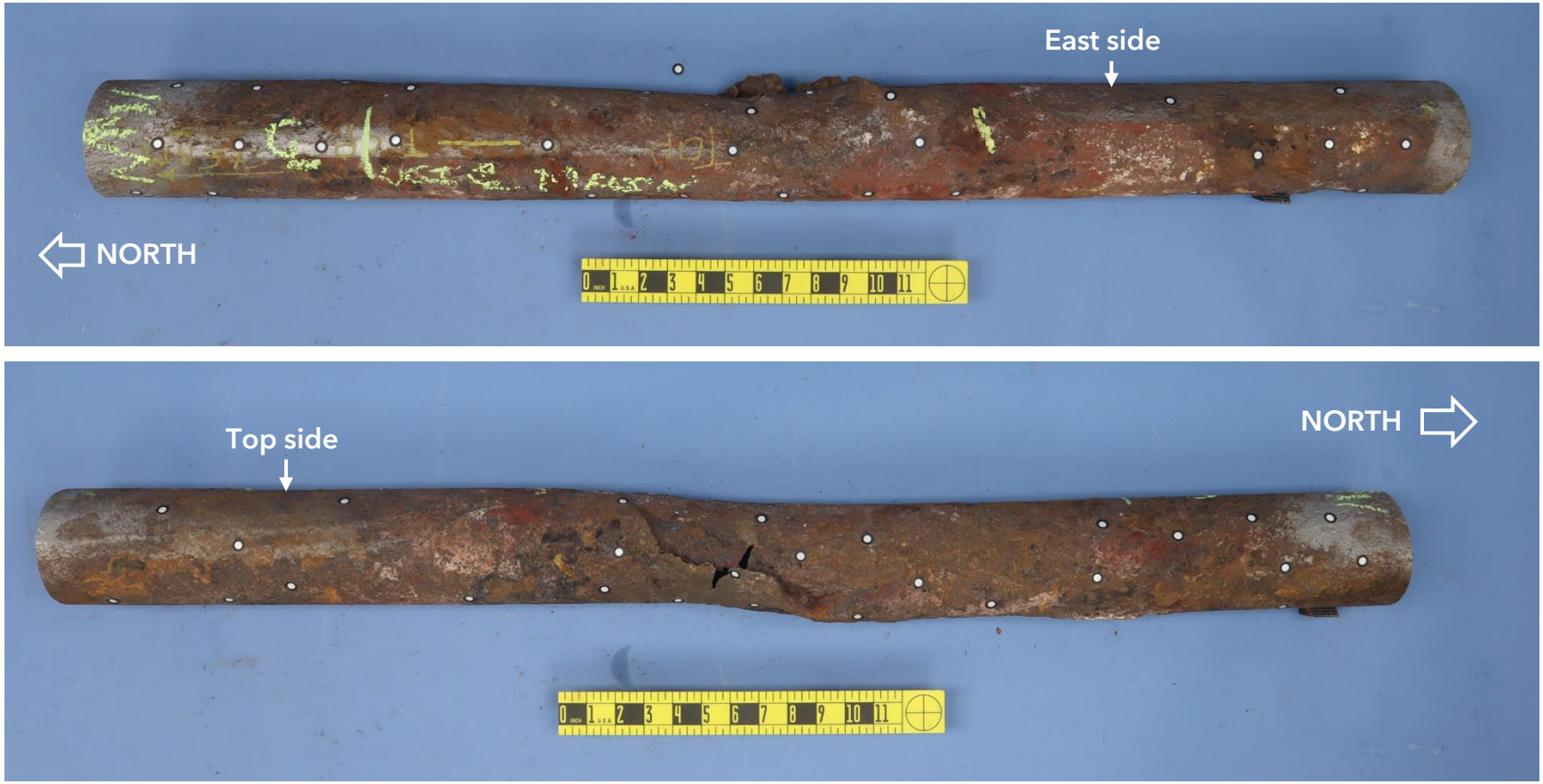


Figure 21. Images of the segment of steam pipe: Top: Top down view; Bottom: East side view.

a)

b)

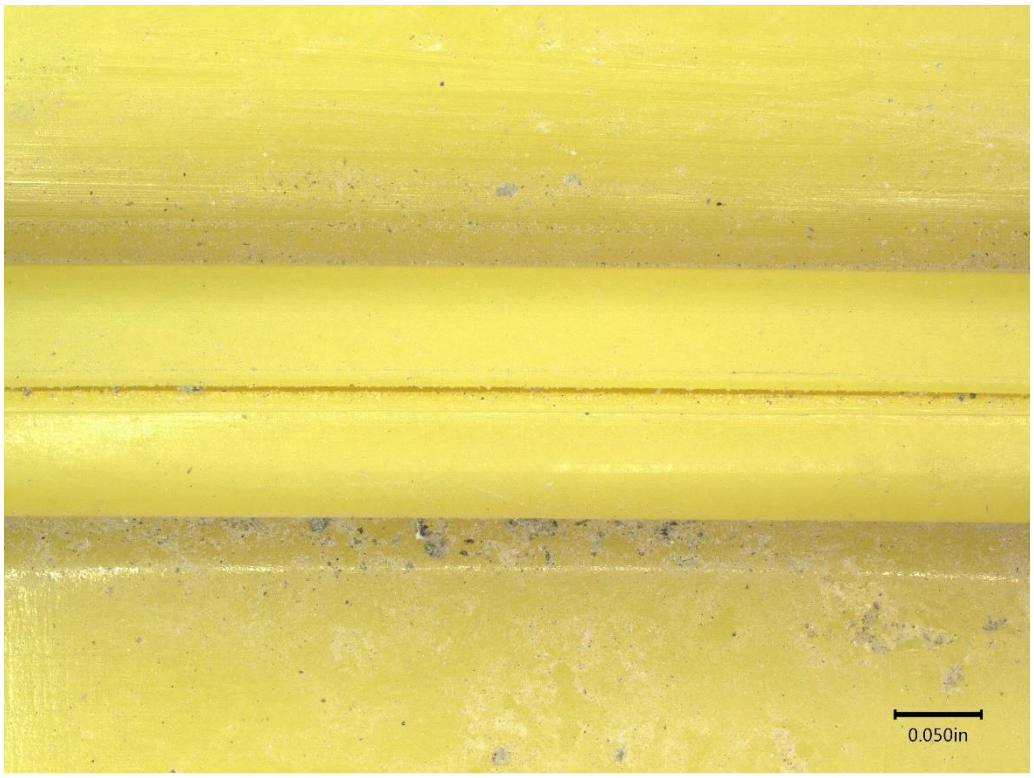
Figure 22. Images of the breach in the steam pipe: a) viewed from the side and b) viewed down the axis of the outward buckle in the pipe wall.

a)

b)

Figure 23. Images of the collapsed in-service marker ball: a) first side and b) opposite side.

a)



b)

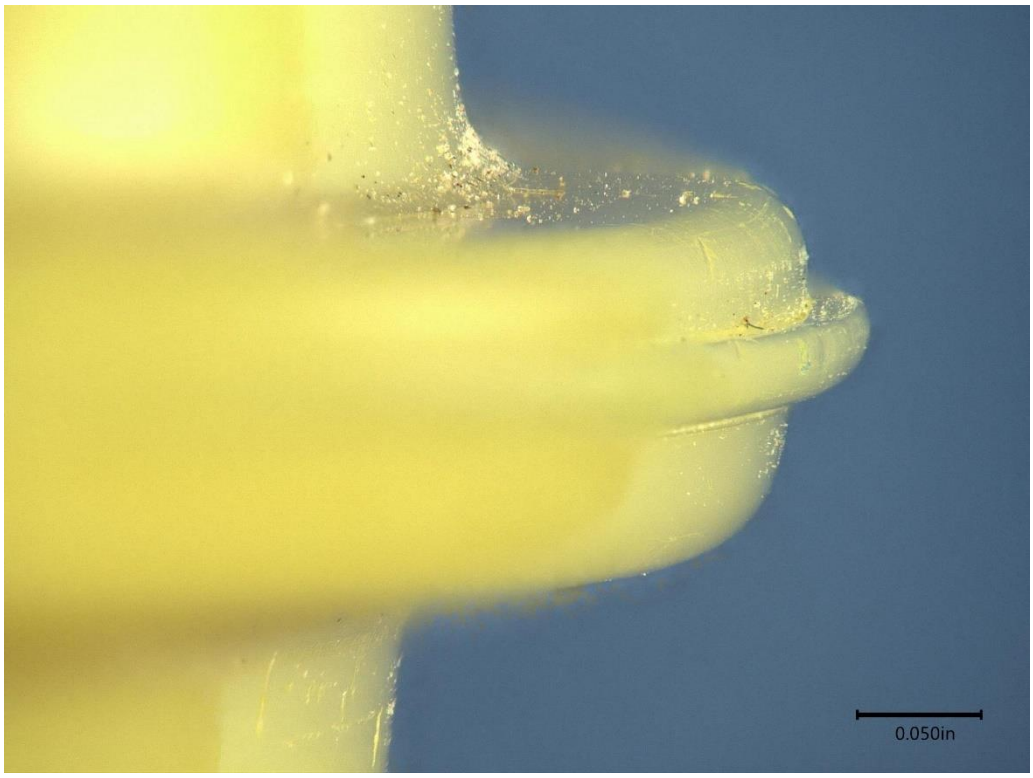
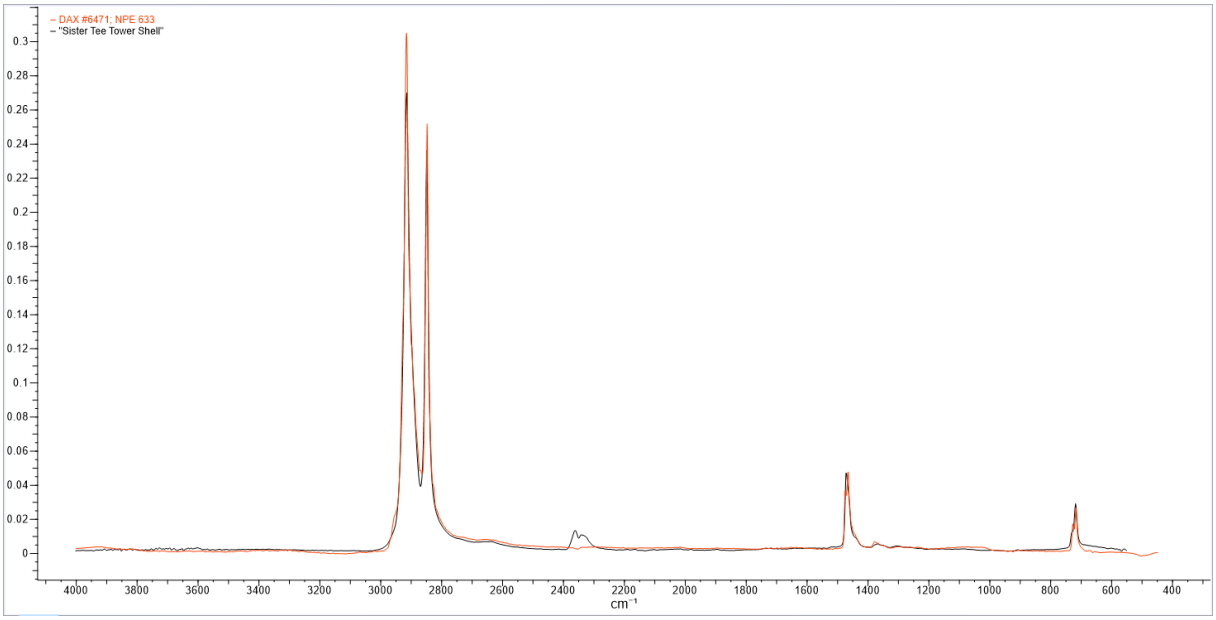


Figure 24. a) Seam of in-service marker ball with missing weld bead and b) exemplar marker ball with intact weld bead.

Figure 25. Sister tee from 77 S. 2nd Ave after removal of the cap showing grease on the insert and cutter.

a)



b)

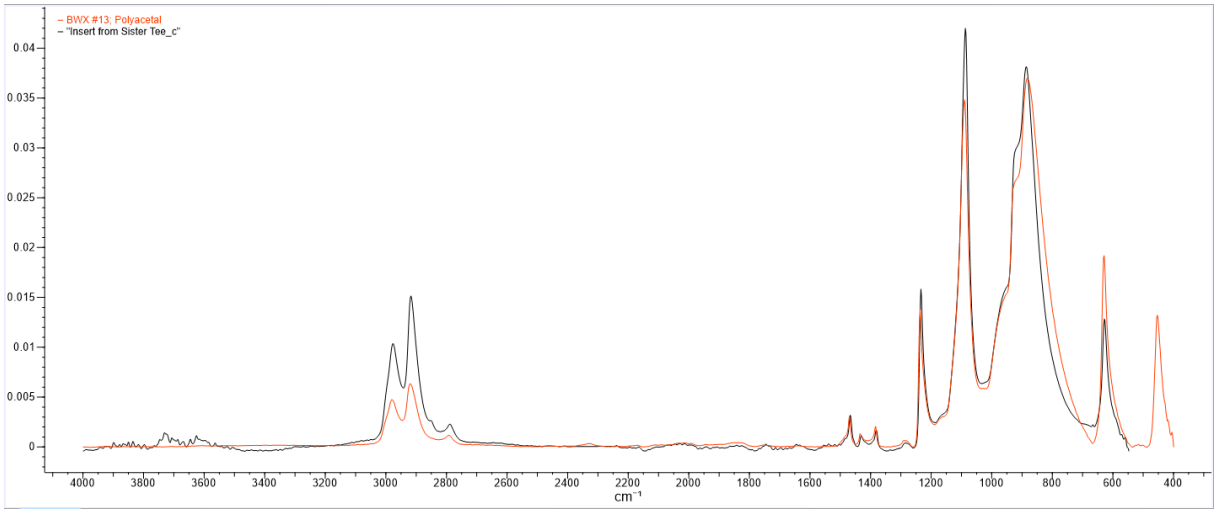
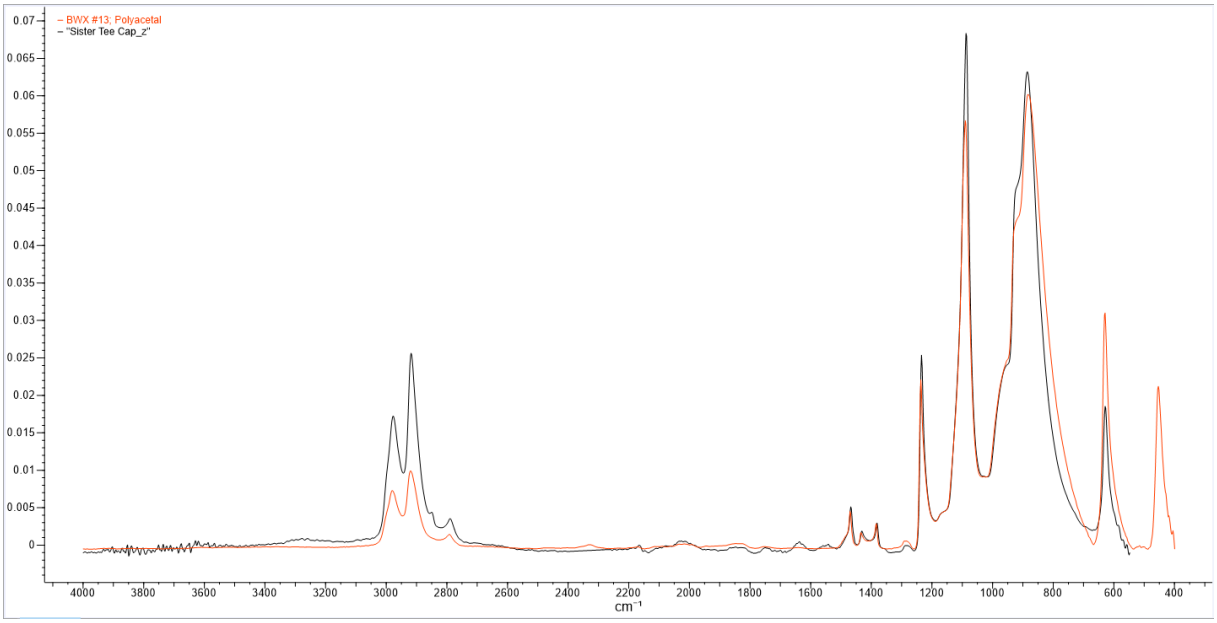


Figure 26. FTIR ATR spectra from sister tee components: a) polyethylene shell; b) polyoxymethylene (polyacetal) insert;

c)



d)

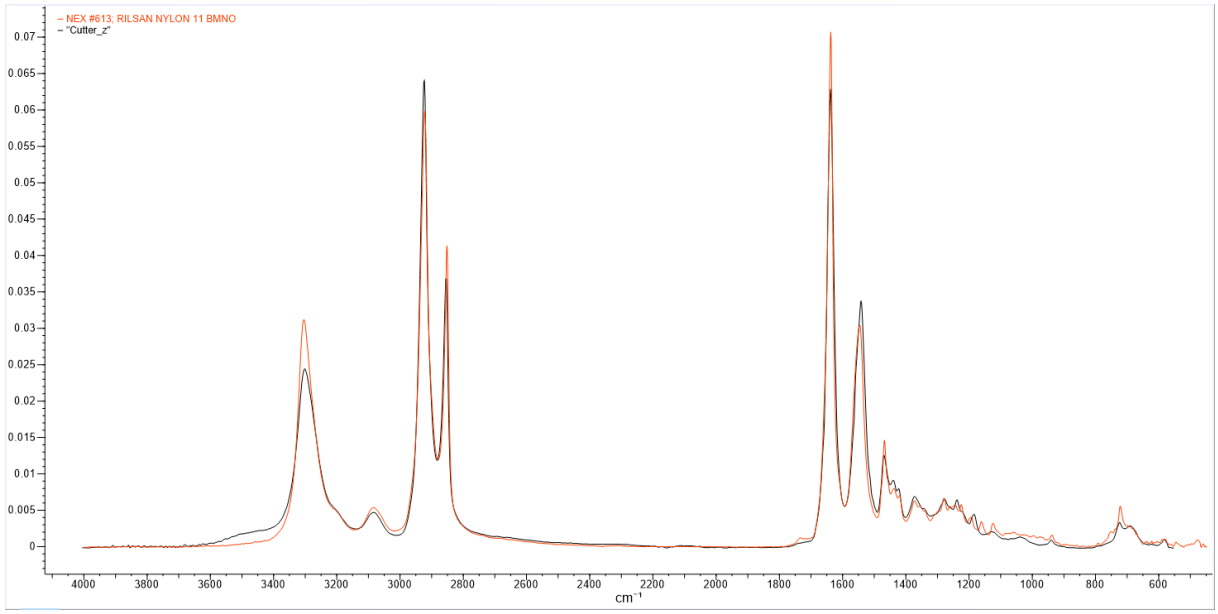
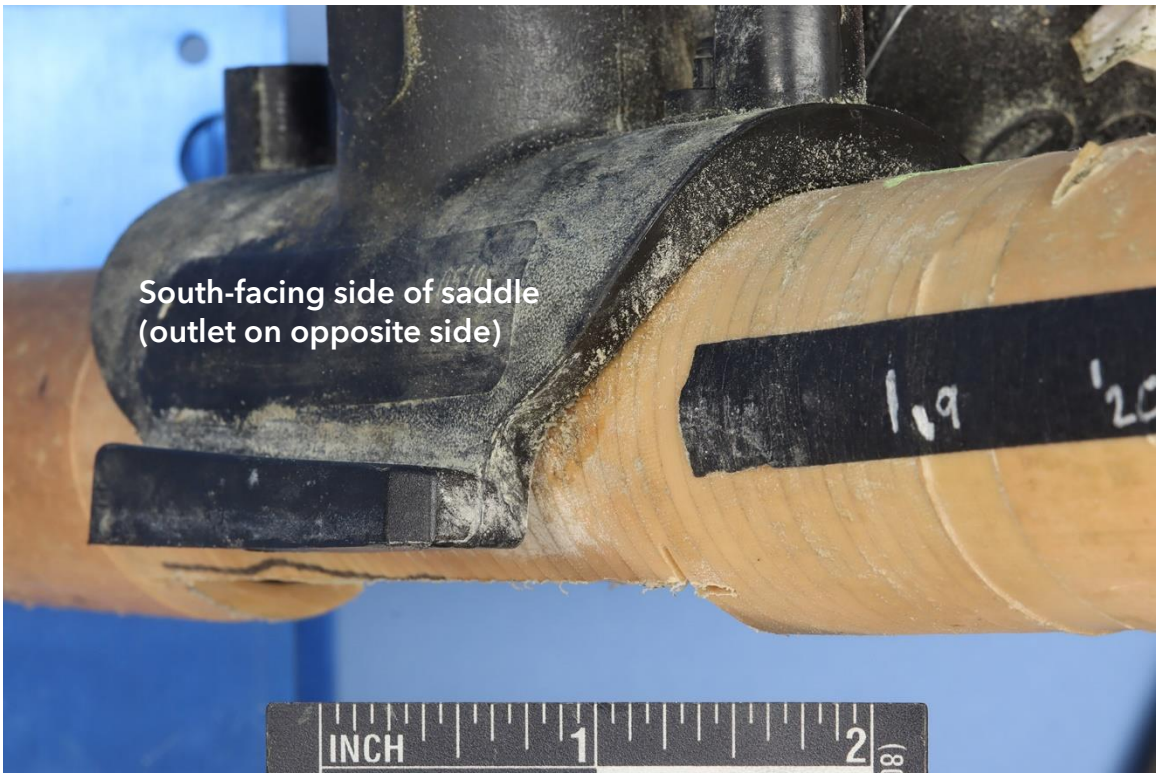


Figure 26 (cont.). c) polyoxymethylene (polyacetal) cap; and d) polyamide cutter head.

a)



b)

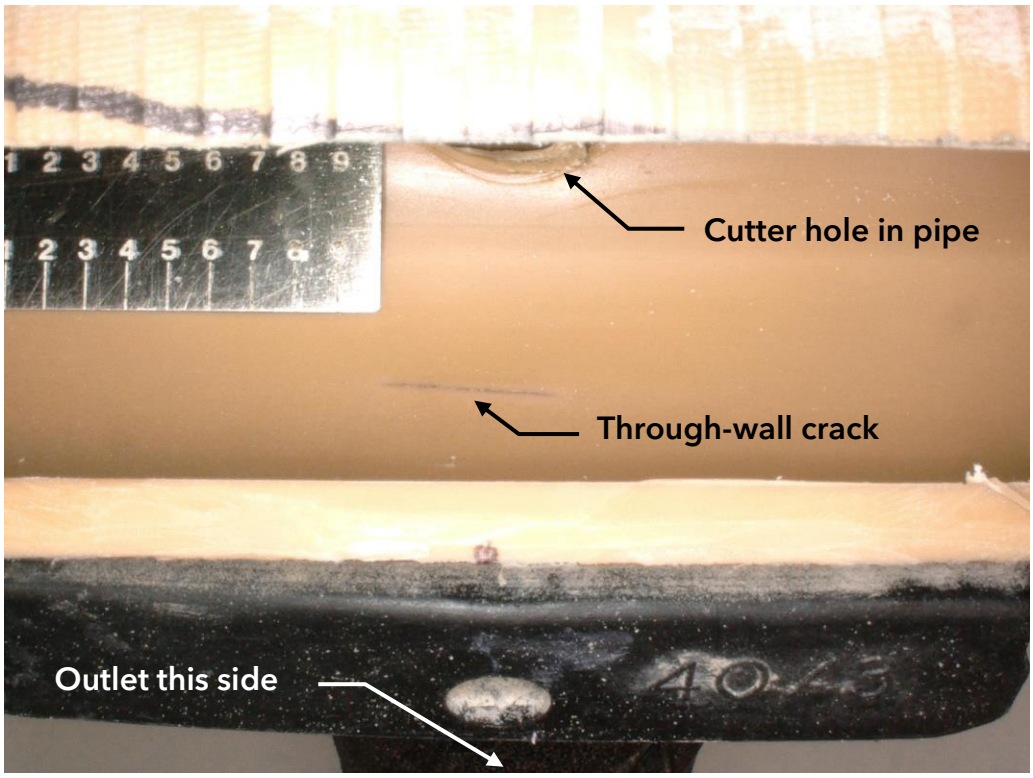


Figure 27. a) Image of 2021 replacement tee with undersaddle and pipe section removed and b) image of the pipe ID surface showing the through-wall crack.

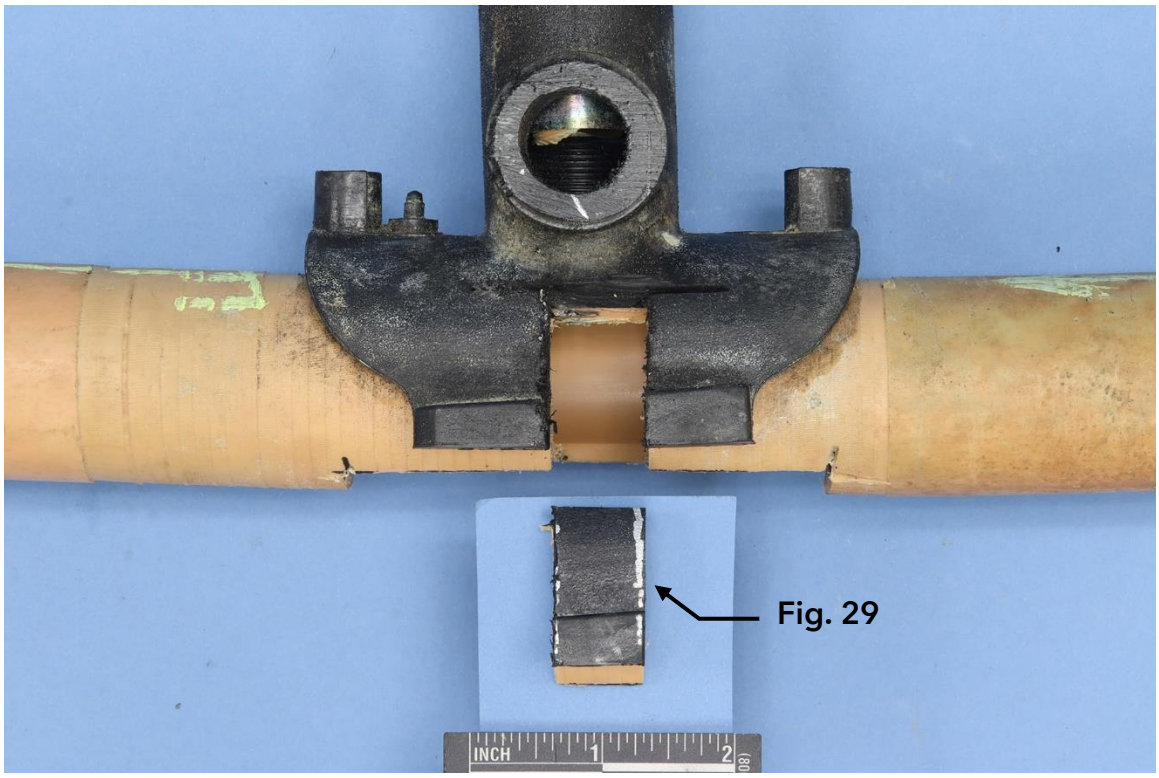


Figure 28. Image showing the piece of saddle and pipe material removed to expose the through-wall crack.

Figure 29. Cross section view of the joint between the saddle and the main pipe.

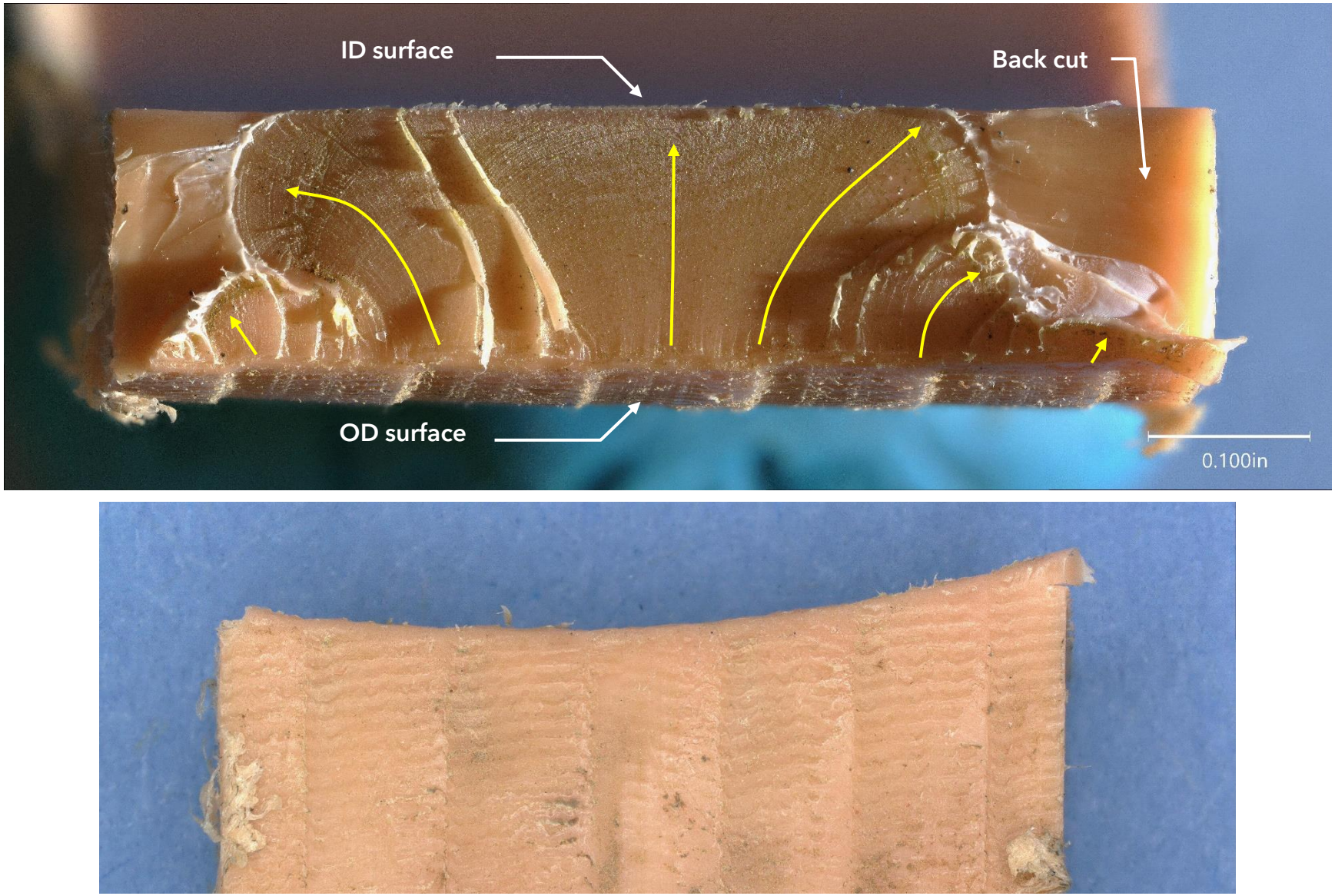


Figure 30. Top: Fracture through the wall of pipe associated with the 2021 replacement teel. Bottom: side view of fracture on OD surface.

Figure 31. Head of cutter from retired tee.

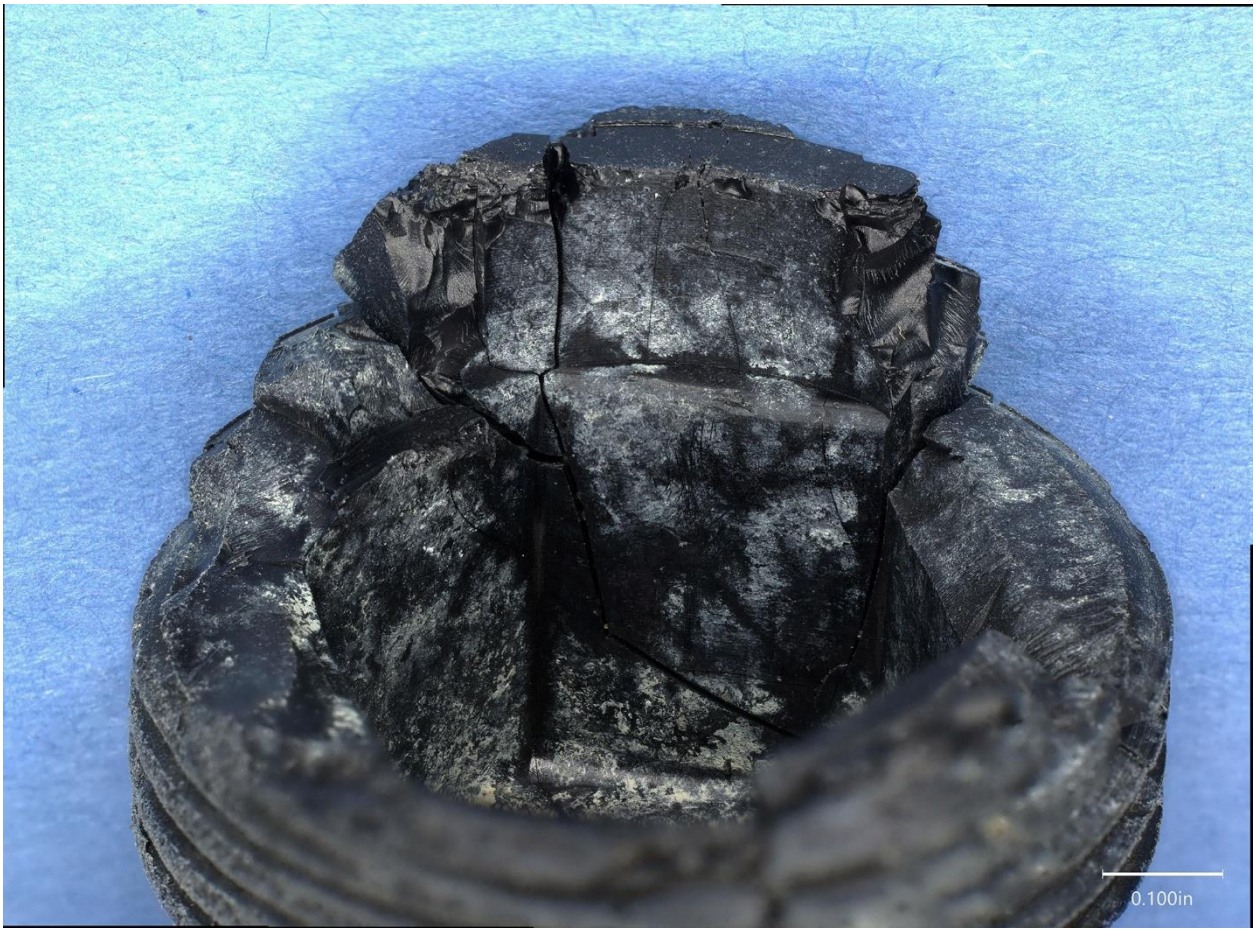


Figure 32. Image showing multiple cracks in the retired tee cutter head.

Figure 33. Image of cutter barrel and threads showing roughening of the surface and rounding of the edge profile.

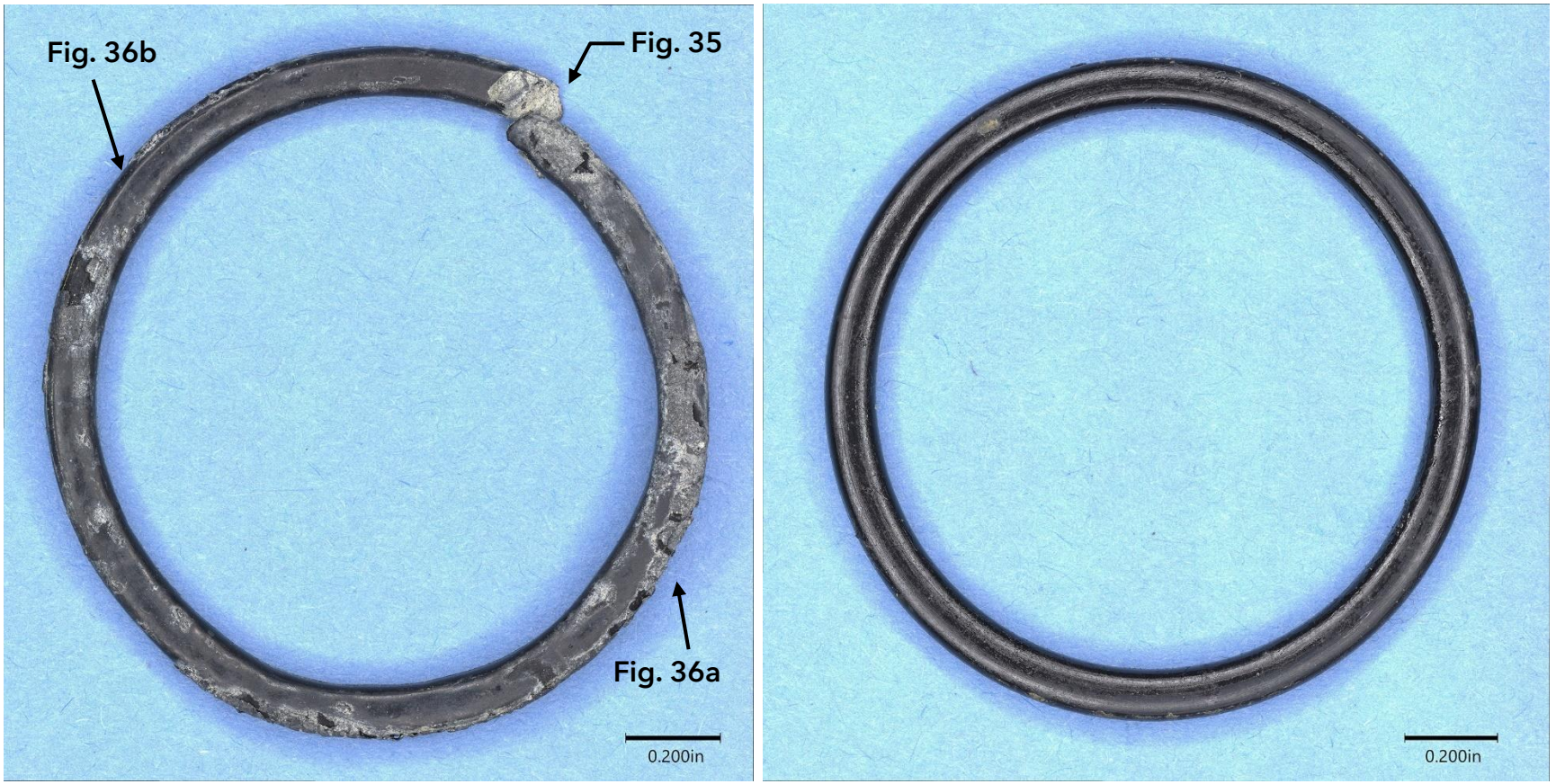


Figure 34. Left image: O-ring recovered from the 17 S. 2nd Ave service tee excavation site. Right image: O-ring from the sister tee providing gas service to 77 S. 2nd Ave.

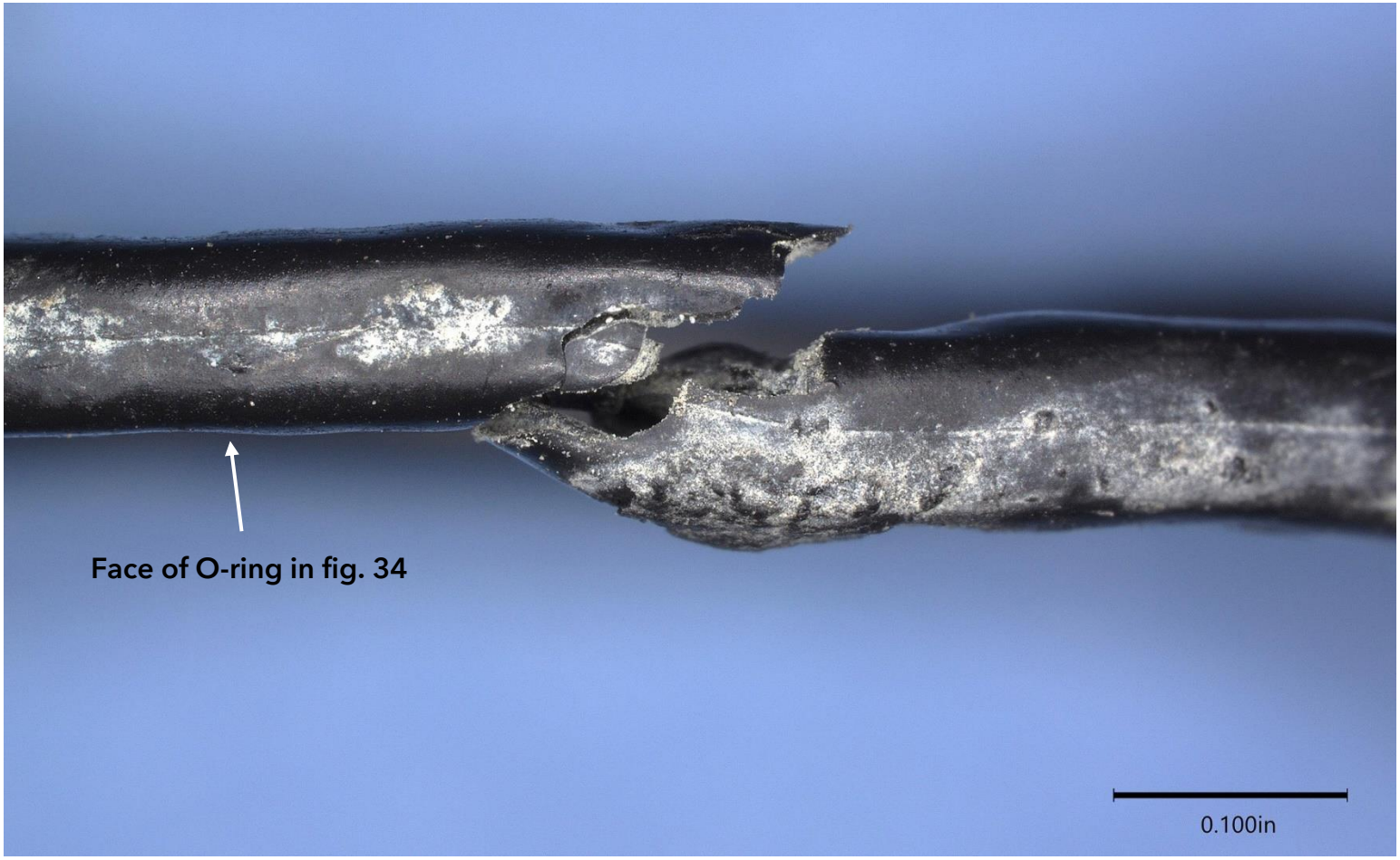


Figure 35. Image of the split in the recovered O-ring. The ring was fractured at an oblique angle. There was a bulge/extrustion on one side of the ring at the fracture location.

a)

b)

Figure 36. Images showing degradation of the recovered O-ring material: a) location 90° clockwise from the split as shown in figure 34 and b) location 90° counterclockwise from the split as shown in figure 34.

APPENDIX A – EXAMINATION OF 17 S. 2ND AVE SERVICE LINE

During pressure testing on scene, the 17 S. 2nd Ave service line did not hold pressure. The service line and riser were submitted to the laboratory for examination as shown above in figure 4a. The 1" CTS service line entered a Perfection Corp. 1-1/4" flexible riser. The downstream end of the riser was connected to a rigid steel tube riser that led to the meter. The main body of the flexible riser was composed of a coiled steel strip covered by a rubber sleeve. A moisture seal covered the upstream end where the service line entered the riser, and a steel fitting connected the downstream end of the flexible riser to the rigid riser tube. The coiled steel strip and rubber sleeve had pulled away from the downstream fitting exposing the 1" service line, as shown in figures A1 and A2. The fitting at the downstream end exhibited signs of plastic deformation. The exposed 1" service line was deformed and partially flattened/ovalized. At one spot, the service line had impinged on the deformed end of the fitting and the wall of the service line had been breached, as indicated in figures A2 and A3 (Note: The fitting on the flexible riser was unthreaded from its downstream connection to the rigid pipe, causing rotational scoring to the service line in the process). The features were consistent with a breach of the service line due to mechanical damage by and to the flexible riser.

Figure A1. Closer view of flexible riser downstream end showing separation of coiled winding and deformation to end fitting.

Figure A2. Image of the breach in the 1-inch plastic pipe, cut by the exposed end of the downstream end fitting.

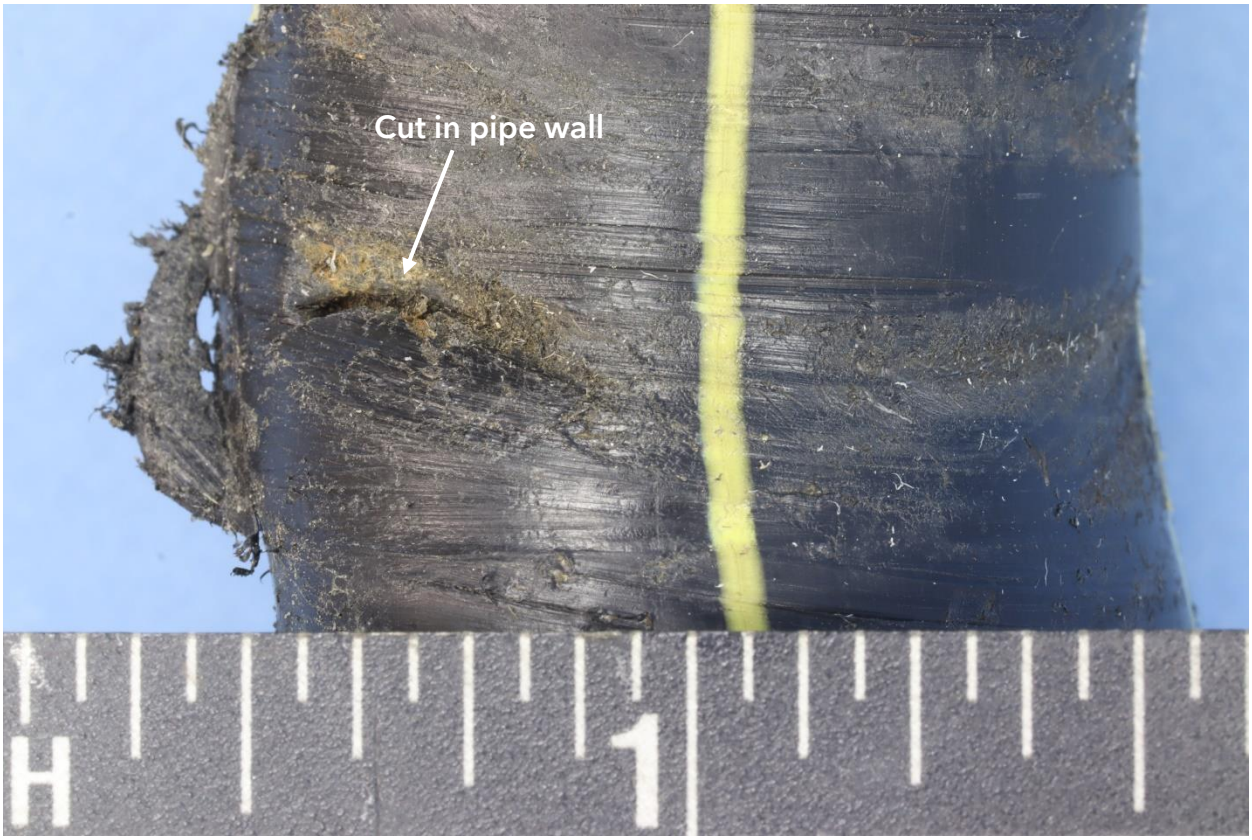


Figure A3. Image showing cut in 1 inch service line.

APPENDIX B – PIPE MARKINGS ON THE 1-1/4" ALDYL A GAS MAIN

The segments of Aldyl A gas main adjacent to the 17 S. and 77 S. 2nd Ave service tees were examined for pipe markings. The ASTM 2513 designation was observed but the material designation was not. An approximately 31-inch length of 2-inch steel pipe with 1-1/4" Aldyl A gas main inside, originally located just upstream of the 17 S. 2nd Ave service tees (retired tee and active tee), was included with the other components. The Aldyl A gas main was removed from the steel pipe by inserting an appropriately sized punch in one end of the steel pipe and tapping the end of the Aldyl A pipe until the two pieces were separated. The markings were examined with the aid of a stereo microscope. The material marking, shown in figure B1 and digitally enhanced in figures B2a and B2b, was faint but was identified as PE 2306 AF BE CD.

The 2-letter suffix codes were interpreted with the aid of ASTM B2513 – 78ES. Table X1, excerpted from that standard, is shown in figure B3. According to the standard, the two letter codes are read as a maximum temperature of applicable rating followed by a hydrostatic design basis at that temperature. For example, the AF code is read as a maximum temperature of 38 °C (100 °F) and a corresponding hydrostatic design basis at that temperature of 1250 psi.

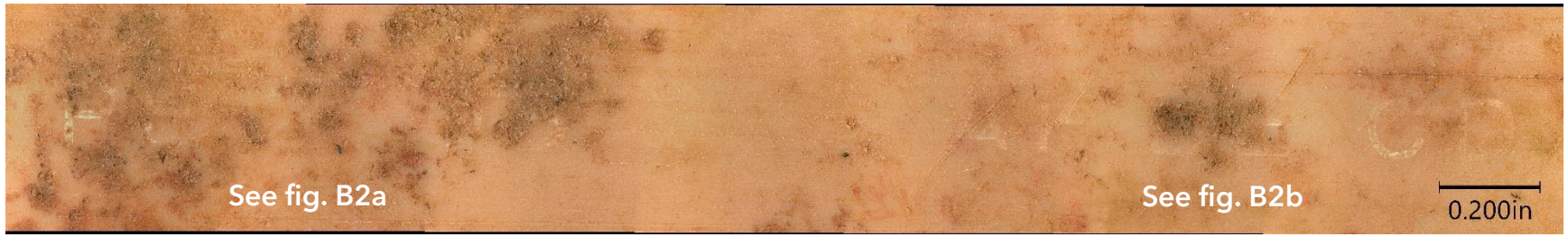


Figure B1. Original image of pipe material marking found on the 1-1/4-inch Aldyl A gas main. Digitally enhanced images of the markings are shown in figures B2a and B2b.

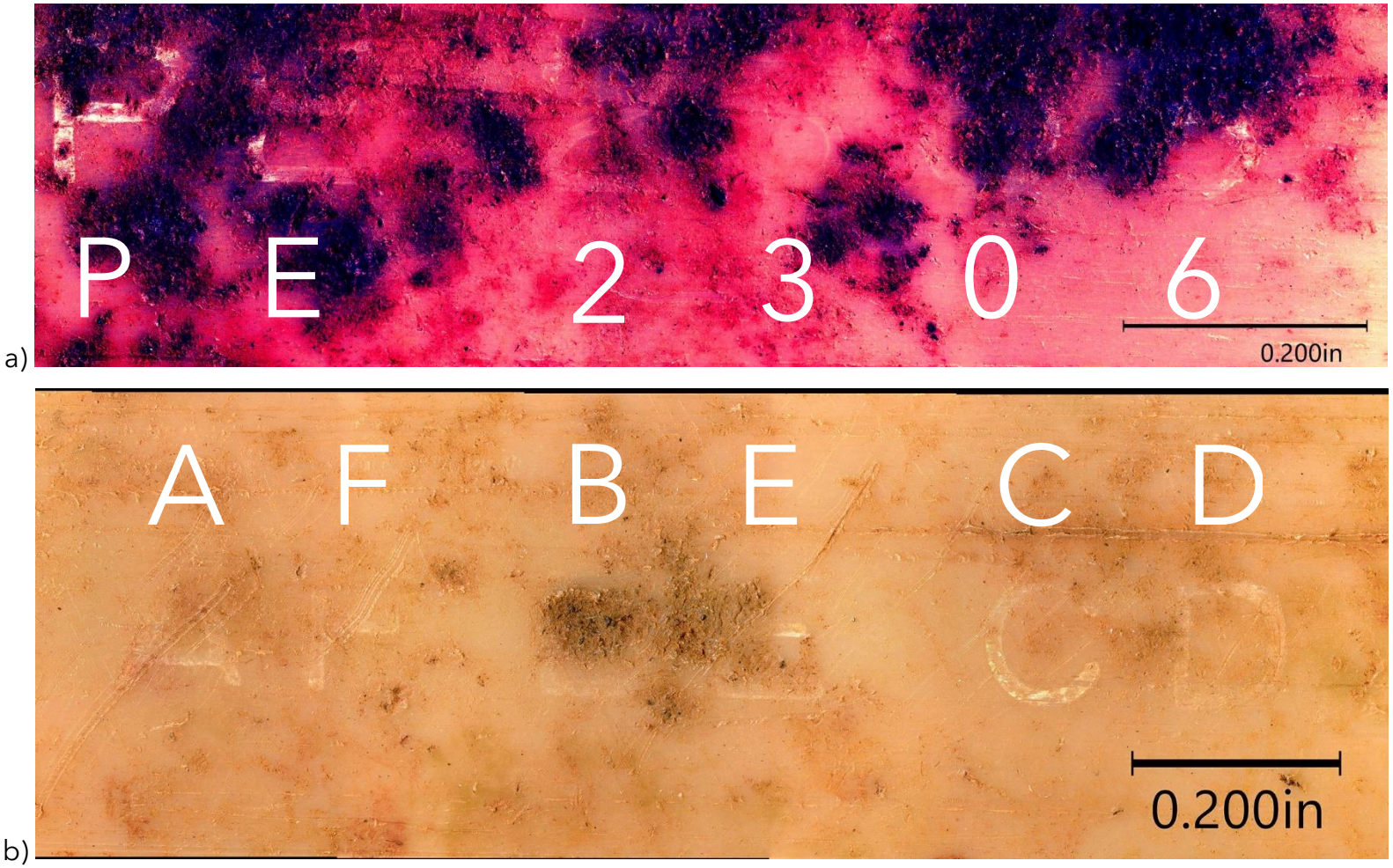
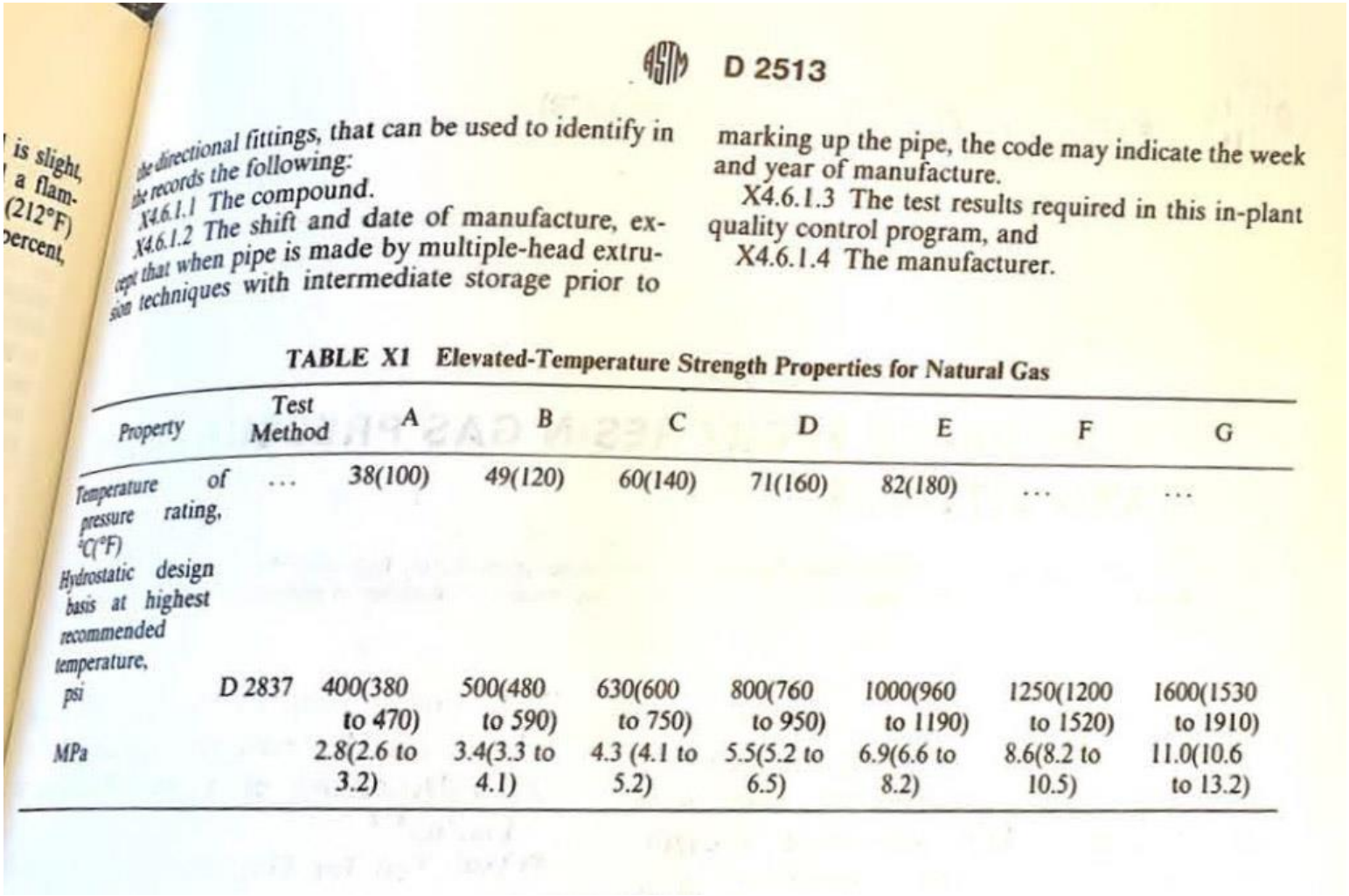


Figure B2. Digitally enhanced images of the pipe markings: a) PE 2306 marking and b) AF BE CD marking.



D 2513

directional fittings, that can be used to identify in records the following:

X4.6.1.1 The compound.

X4.6.1.2 The shift and date of manufacture, except that when pipe is made by multiple-head extrusion techniques with intermediate storage prior to marking up the pipe, the code may indicate the week and year of manufacture.

X4.6.1.3 The test results required in this in-plant quality control program, and

X4.6.1.4 The manufacturer.

TABLE X1 Elevated-Temperature Strength Properties for Natural Gas

Property	Test Method	A	B	C	D	E	F	G
Temperature of pressure rating, °C(°F)	...	38(100)	49(120)	60(140)	71(160)	82(180)	...	...
Hydrostatic design basis at highest recommended temperature, psi	D 2837	400(380 to 470)	500(480 to 590)	630(600 to 750)	800(760 to 950)	1000(960 to 1190)	1250(1200 to 1520)	1600(1530 to 1910)
MPa		2.8(2.6 to 3.2)	3.4(3.3 to 4.1)	4.3 (4.1 to 5.2)	5.5(5.2 to 6.5)	6.9(6.6 to 8.2)	8.6(8.2 to 10.5)	11.0(10.6 to 13.2)

Figure B3. Table X1 from ASTM D-2513 – 78ES showing table for decoding 2-letter material suffix codes. The first letter indicates the maximum temperature of the applicable rating and the second letter indicates the corresponding hydrostatic design basis at that temperature.

APPENDIX C – EXTERNAL LABORATORY EXAMINATION REPORT

Plastic Material Testing & Analysis for NTSB

ESi Project No: 99846

Report Prepared For

Donald Kramer
NTSB
490 L'Enfant Plaza East, SW
Washington DC, 20594

Submitted by:

[redacted]

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Principal
Director of Polymeric, Composite & Non-Metallic Materials Practice
Illinois P.E. | Expires: November 30, 2023

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062.058788
STATE OF ILLINOIS

September 27, 2023
Date

Technical Review by:

[redacted]

Gaurav Nagalia
Senior Staff Consultant

September 27, 2023
Date

Phone: 630-851-4566 | Fax: 630-851-4870 | Toll Free: 866-596-3994

www.engsys.com

Introduction

National Transportation Safety Board (NTSB) retained Engineering Systems Inc., (ESi) to perform plastic material testing and characterization on components from subject and exemplar Aldyl A Tapping Tee fittings. There were three specific material testing requests as follows:

1) Thermal analysis utilizing differential scanning calorimeter (DSC) on a white Polyoxymethylene (POM) insert after it is removed from the shell of the exemplar tee fitting to determine if the POM insert is consistent with Delrin® acetal resin based on peak melting temperature.
2) Fourier Transform Infrared (FTIR) microscopy using micro-FTIR technique on a microtome section of the MDPE material near the longitudinal fracture to determine if any oxidative degradation is present in the polyethylene material.
3) Gas Chromatography and Mass Spectroscopy (GC-MS) testing to evaluate the presence of any organic volatile chemicals absorbed in the POM insert from service by comparing the results of GC-MS of subject POM insert with GC-MS results obtained on POM insert from exemplar Tee fitting.

Based on the information provided, the subject and exemplar Aldyl A tapping tee fittings were components from NTSB's investigation into the natural gas-fueled explosion and fire that occurred at Building 2 of the R. M. Palmer Company in West Reading, Pennsylvania. Donald Kramer, materials engineer from NTSB, hand carried the fitting samples for the analysis to ESi labs located in Aurora, IL on September 6, 2023. The sample preparation was performed in presence of Donald Kramer of NTSB as well as Mark Connors of UGI Utilities, Inc. ESi only utilized small fragments from the subject and exemplar fitting samples; all other remnants were always in the chain of custody of Donald Kramer of NTSB. The sign-in sheet documenting the attendance during ESi's sample preparation for the testing is provided in Appendix A. Digital photos were taken to document the work effort and a file share link will be provided along with this report for you to download all the digital images for your file.

This report provides the summary of the work ESi performed for this engagement.

Material Testing and Characterization

1) DSC Analysis on POM material (POM) insert after it is removed from the shell of the exemplar tee fitting.

DSC Analysis was performed using Thermal Analysis (TA) Instruments Differential Scanning Calorimeter (DSC Q2000). In a DSC analysis the samples are heated at a controlled heating rate (20 °C/minute) and the heat differential required to maintain an empty sample pan and sample pan with sample is used to detect thermal transitions in polymeric materials. DSC is used to assist in identifying specific polymers based on thermal transition temperatures. The test data obtained using the DSC is provided in Appendix B of this report.

The experiment was conducted by heating the sample, cooling the sample, and re-heating the sample as described in ASTM D 3418-21 titled "Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential scanning Calorimetry". The peak melting temperature was found to be in the range of 175-177 degrees Celsius. *The DuPont product and properties Guide for Delrin® acetal resin (published 2000)* reports the melting temperature of all general-purpose grades of Delrin as 178 degrees. Based on the peak melting temperature of the DSC analysis, the material insert was consistent with a polyoxymethylene (POM) homopolymer and consistent with Delrin®, a DuPont registered trademark for its brand of acetal resin also commonly referred to as POM.

2) Fourier Transform Infrared (FTIR) microscopy using micro-FTIR technique on a microtome section of the MDPE material near the longitudinal fracture.

Micro-FTIR spectroscopy was utilized to profile the degree and depth of oxidation in the MDPE material at the location of the fracture in the subject Aldyl A tee fitting. Infrared spectroscopy was utilized as early as the 1950's as a technique by which oxidation could be identified in polyethylene materials. A parameter called the "carbonyl index" was created to quantify the extent of degradation.

The carbonyl index was defined as the intensity of the infrared absorption peak of a specific carbonyl moiety formed in the oxidation of HDPE material normalized by the intensity of a methylene absorption peak in the same spectrum. These carbonyl groups that result from oxidation of polyolefin materials have characteristic infrared absorption frequencies. Among these groups, the strongest absorption peak is observed at about 1710 to 1720 cm^{-1}. When oxidation occurs, a peak near 1710 cm-1 to 1720 cm-1 is formed, which progressively increases in intensity as the degree of polymer oxidation increases. The carbonyl index is defined as the ratio of this carbonyl absorbance to that of the polymer absorption band at approximately 1465 cm^{-1}. The use of this ratio compensates for any differences in sample thickness and serves as an internal standard.

The carbonyl index was profiled through the thickness of the sample in order to determine the extent of any oxidation, if it exists. This profiling is possible with a Micro-FTIR instrument, which allows one to focus the infrared beam at a precise location on the sample. The samples consist of microtomed cross-sections of the tubing wall that ranged from 0.0005 to 0.001 inches in thickness. The thickness of the microtomed specimens was sufficiently small so that all absorbance measurements were in the detector linear absorption range. The infrared spectra were recorded in 0.03 mm increments (~0.0012 inches), using a 0.030 mm deep x ~0.300 mm wide aperture and starting at the inner surface. The profiling was continued until the carbonyl absorbance approached the center. The results are summarized in Appendix C of this report.

There was no apparent absorbance in the carbonyl region. Thus, there was no need to calculate a carbonyl index.

3) Gas Chromatography and Mass Spectroscopy (GC-MS) testing

Solid Phase Microextraction (SPME) gas chromatography and mass spectroscopy analysis was performed on the POM insert material from the subject Aldyl A tee as well as from exemplar subject Aldyl A tee. The testing was performed using a PE Clarus 600/600D GC-MS instrument, using a 30 m x 0.25 mm I.D. fused silica capillary column. The details of the procedure are provided along with the full report in Appendix D. The SPME GC/MS analysis detected very little low molecular weight organic compounds in each sample. The subject POM insert material contained less detectable compounds than exemplar material.

<<< End of Report Text >>>

Appendices:

Appendix A: Sign-In sheet

Appendix B: 99846-D001-Sister Insert-001 heat cycle 1
99846-D001-Sister Insert-001 heat cycle 3

Appendix C: 99846-F001-Inner Surface
99846-F001-Outside Surface

Appendix D: H0712 MEi Report for ESI 99846

Appendix A

Sign-in Sheet

INSPECTION SIGN-IN

NTSB / PLASTIC MATERIAL TESTING & ANALYSIS

ESi Matter #: 99846

Date: September 6, 2023

(Please Print)

Please leave a business card if possible

↑ Name

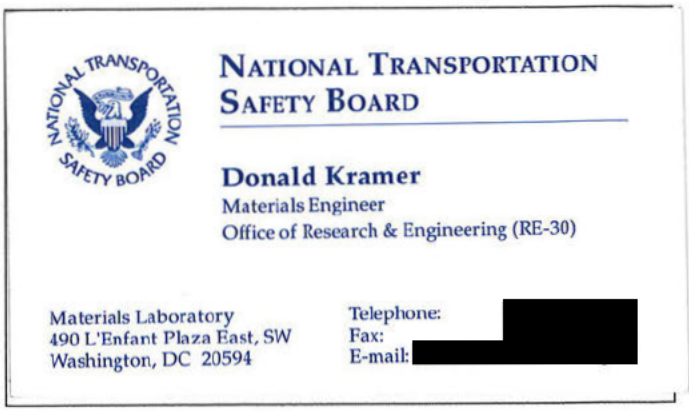
↑ Company

NTSB

↑ Representing

[redacted]

↑ Signature



↑ Name

↑ Company

UGI UTILITIES, INC.

↑ Representing

[redacted]

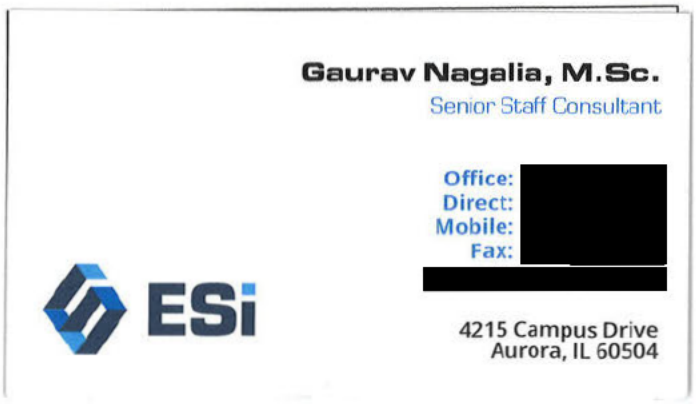
↑ Signature

↑ Name

↑ Company

↑ Representing

[redacted]



By signing this form, you acknowledge that you are visiting ESi and agree to fully cooperate with ESi staff relative to security, safety, and confidentiality. You further agree not to photocopy or videotape any items without permission of ESi Management and indemnify and agree to hold ESi harmless in the event of any loss.

INSPECTION SIGN-IN

NTSB / PLASTIC MATERIAL TESTING & ANALYSIS

ESi Matter #: 99846

Date: September 6, 2023

(Please Print)

Please leave a business card if possible

A. Shah
↑ Name

ESi
↑ Company

↑ Representing [redacted]

↑ Signature

↑ Name

↑ Company

↑ [redacted]

↑ Name

↑ Company

↑ Representing

↑ Signature

(business card)

By signing this form, you acknowledge that you are visiting ESi and agree to fully cooperate with ESi staff relative to security, safety, and confidentiality. You further agree not to photocopy or videotape any items without permission of ESi Management and indemnify and agree to hold ESi harmless in the event of any loss.

Phone: 630-851-4566 | Fax: 630-851-4870 | Toll Free: 866-596-3994
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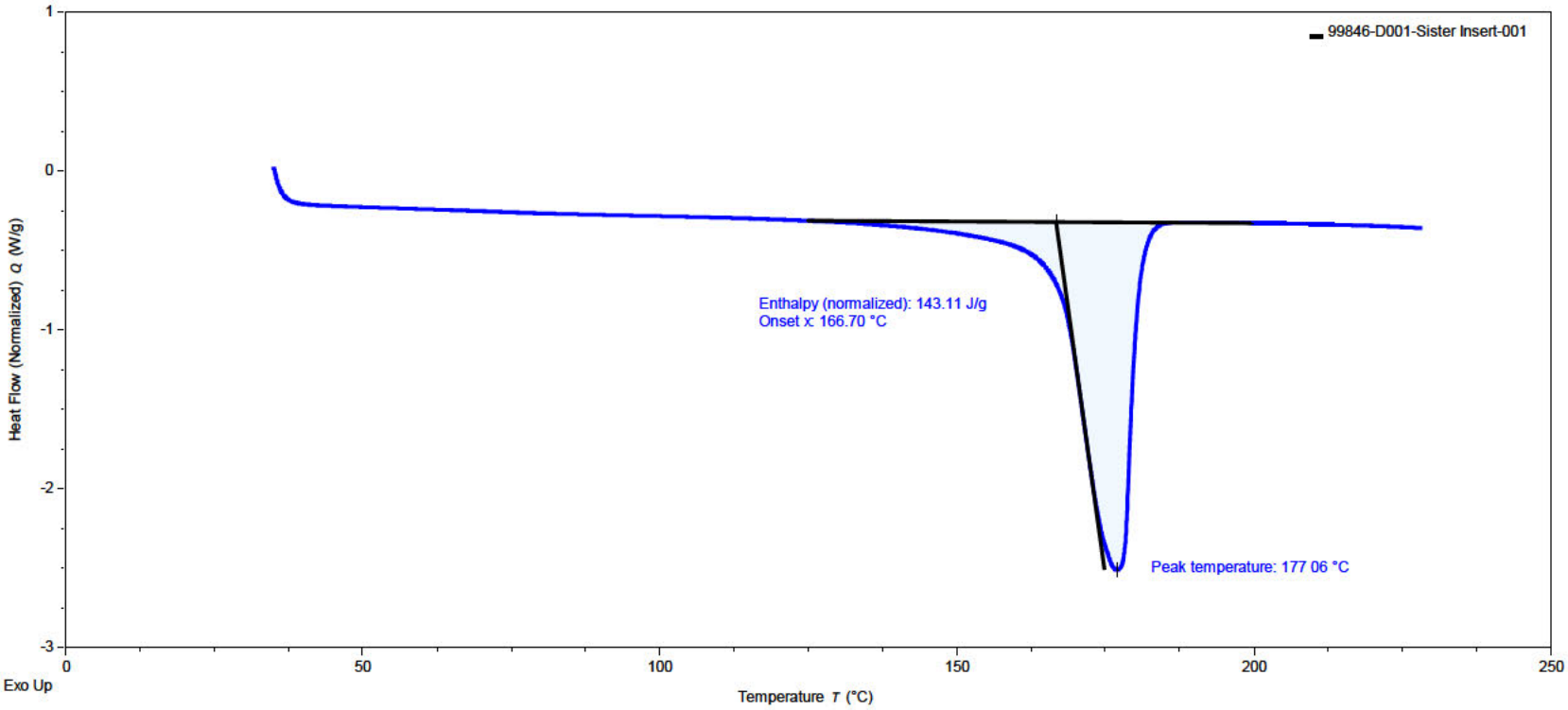
Appendix B

D001-Sister Insert-001 heat cycle 1
D001-Sister Insert-001 heat cycle 3

D001-Sister Insert-001 heat cycle 1

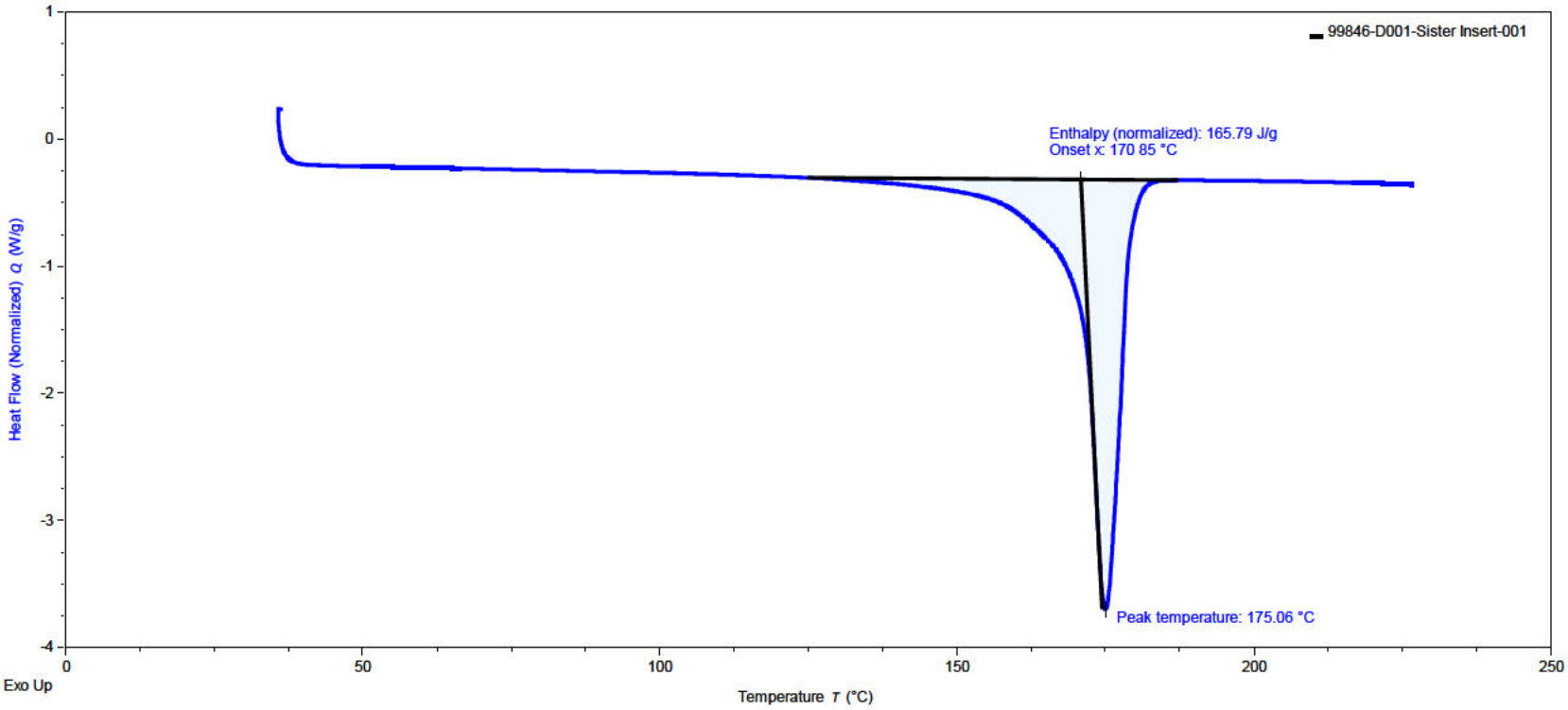
99846-D001-Sister Insert-001



TA Instruments Trios V5.1.1.46572

D001-Sister Insert-001 heat cycle 3

99846-D001-Sister Insert-001

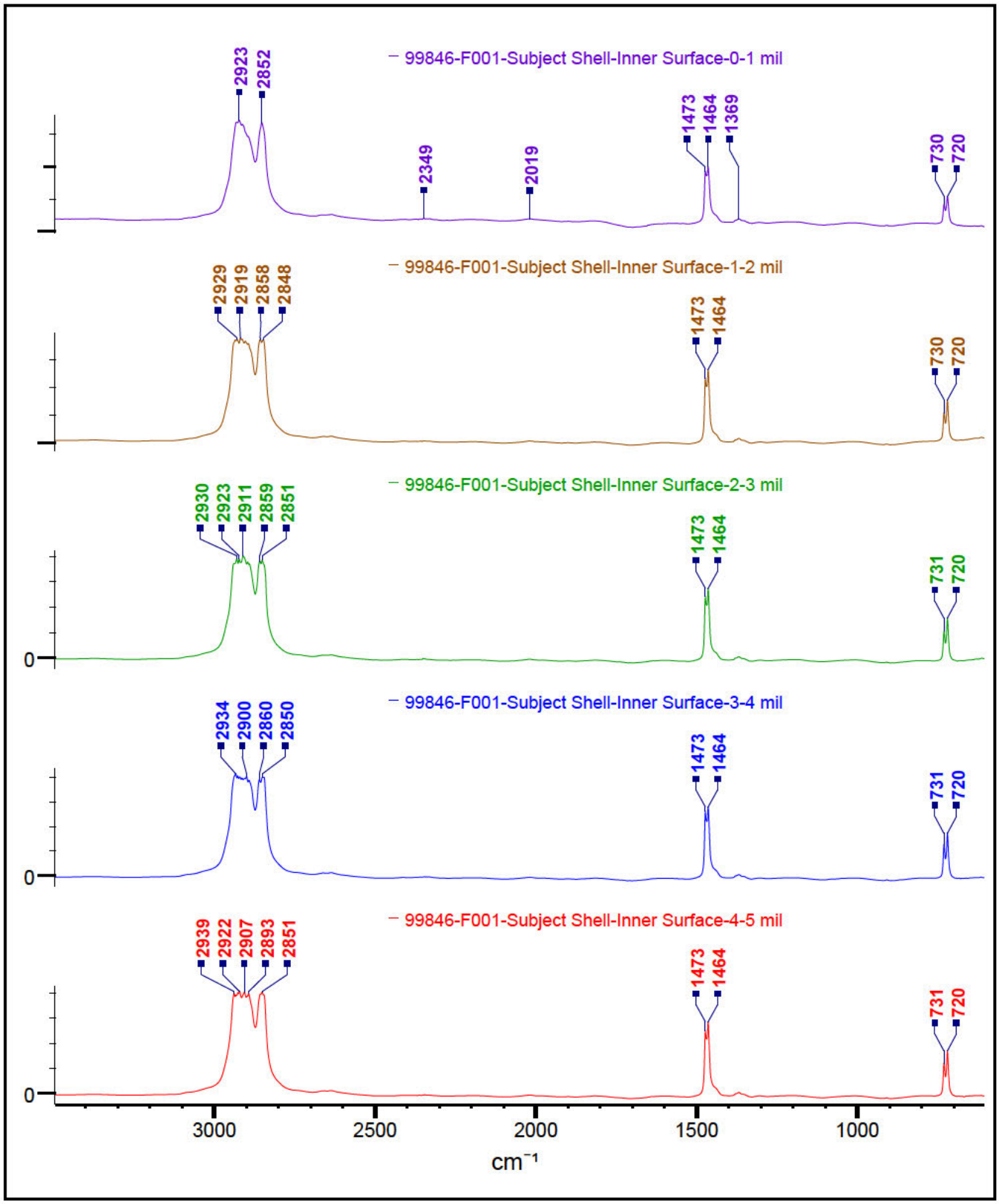


TA Instruments Trios V5.1.1.46572

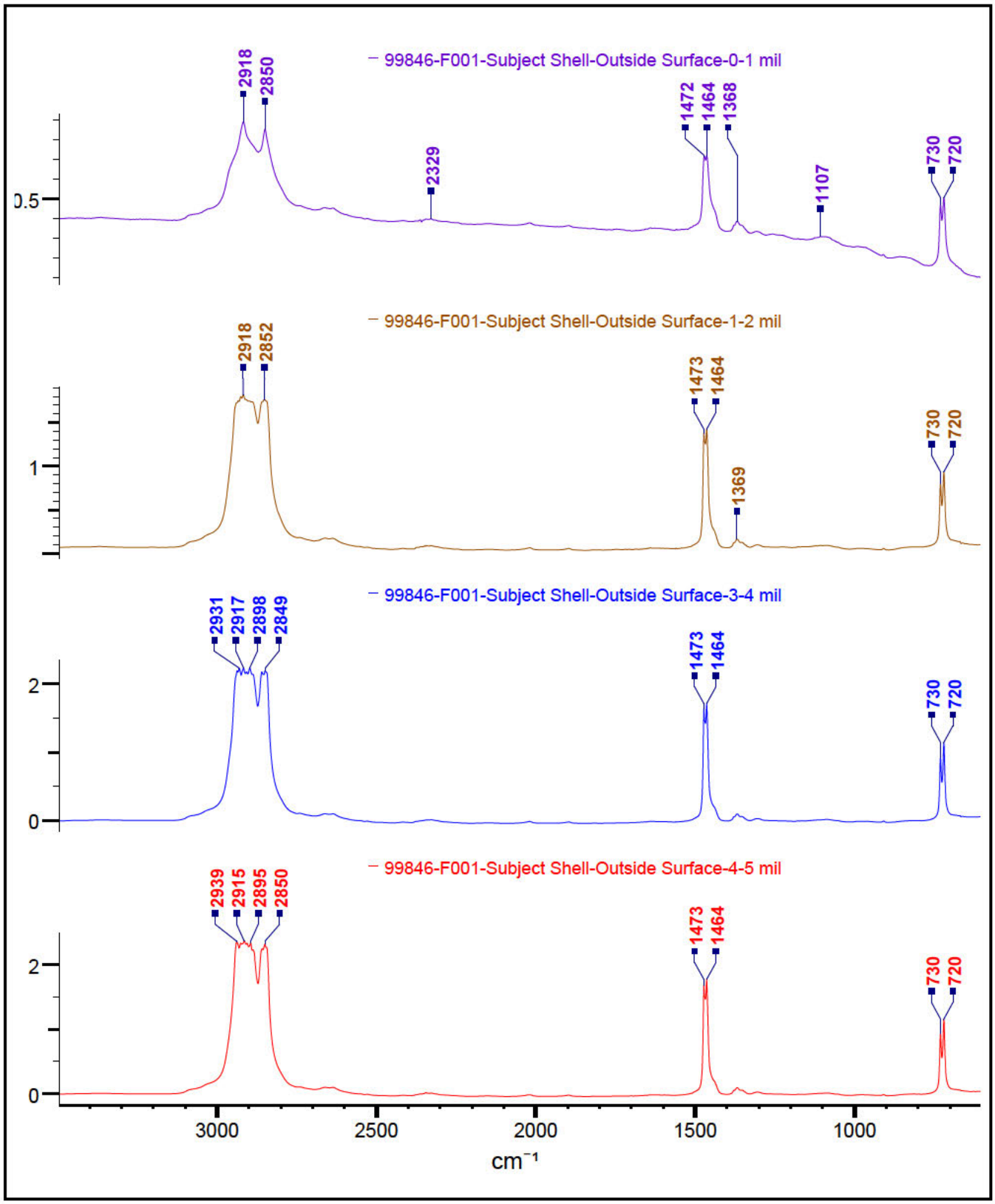
Appendix C

F001-Inner Surface
F001-Outside Surface

99846-F001-Subject Shell Inside Surface Depth Profile FTIR



99846-F001-Subject Shell Outside Surface Depth Profile FTIR



Appendix D

H0712 MEi Report

MEi
MATERIALS
ENGINEERING inc.

47W605 I. C. Trail
Virgil, IL 60151

630/365-9060
FAX 630/365-9138

September 22, 2023

Andy Shah
Engineering Systems, Inc.
4215 Campus Drive
Aurora, IL 60504

Subject: GC/MS Analysis of Two Samples, Project H0712
Reference: P.O. 01-00205A-294-0

Introduction:

Materials Engineering, Inc. was requested to perform solid-phase microextraction (SPME) gas chromatography-mass spectrometry (GC/MS) analysis on two samples associated with Engineering Systems, Inc. Project 99846. The samples are identified as subject and exemplar material. The analysis was conducted to detect any significant amount of low molecular weight volatile compounds. Overall views of the samples are presented in Figure 1.

Procedure:

For the solid phase microextraction (SPME) gas chromatography – mass spectrometry (GC/MS) analysis, an empty vial was placed on a hot plate which had been heated to approximately 61 °C and allowed to stand for five minutes. A 65-µm polydimethylsiloxane-divinylbenzene-Carboxen SPME fiber in a manual holder was inserted into the empty sample vial and exposed to the headspace for thirty minutes so volatile and semi-volatile organic compounds in the headspace could adsorb or absorb onto its coating. The SPME fiber was retracted into the manual holder and removed from the vial. The fiber was immediately transferred to the GC/MS and desorbed in the heated GC/MS injector port at 250 °C for thirty seconds. After the vial had cooled, sample material was placed in the vial and was analyzed twice. In the first trial, the SPME fiber was exposed to the sample for thirty minutes with no heat; in the second trial, the sample was re-exposed for thirty minutes after heating the sample to approximately 61 °C, Figure 2. A new vial was used for each sample.

0.56006 grams of material were used for the subject sample, and 1.03271 grams of material were used for the exemplar sample on 9-8 and 9-9-2023. In addition, a 4.26 milligram sample of dodecane was analyzed in this manner to enable semi-quantitation of detected analytes.

GC/MS analysis was carried out on the PE Clarus 600/600D GC-MS, using a 30 m x 0.25 mm I.D. fused silica capillary column coated with a 0.25 µm phenyl arylene polymer virtually identical to a (5%-phenyl)-methylpolysiloxane stationary phase. The injector temperature was held at 300 °C for the duration of the program. A split ratio of 15:1 was

employed. The column temperature program was as follows: hold 10 minutes at 40 °C; ramp 15 °C per minute to 310 °C; hold 4 minutes at 310 °C. The carrier gas was helium, and a constant flow rate of 1.0 mL/min was used. The mass spectrometer was operated in full scan mode, with a mass range of 19 m/z to 500 m/z.

A total ion chromatogram using the mass range of 19 to 500 m/z was generated for each injection, mass spectra for each peak were compared against the 2017 NIST mass spectral library of approximately 263,000 mass spectra, and compound classes were identified based on the best library match and manual interpretation.

Results:

SPME GC/MS analysis detected very little low molecular weight organic compounds in each sample. The subject material contained less detectable compounds than the exemplar material. The exemplar material did appear to contain traces of alkylbenzenes and polyaromatic hydrocarbons that could be related to fuel or combustion products.

The retention times, compound identification, concentrations and notes regarding the compounds detected in the heated samples are summarized in Tables 1 and 2. The GC/MS chromatograms are presented in Figures 3 through 8. Mass spectra of selected peaks and NIST library search lists are presented in Figures 9 through 19. The dodecane standard analysis results are presented in Figures 20 and 21, while integrated chromatograms from the heated samples are presented in Figure 22.

Please call if you have any questions or require additional analysis.

[redacted]

Steven W. Johnson
Senior Analytical Chemist
[redacted]

Table 1: GC/MS Analysis Results for the Heated Subject Material SPME Sample

Retention Time, Minutes	Type of Compound	Concentration, micrograms detected per gram of sample	Notes
14.46	1-Hexanol, 2-ethyl-	0.3	CAS 104-76-7
15.50	Unknown	0.1	
15.65	Unknown	0.3	
15.89	Siloxane	0.4	
16.73	Alkane	0.1	
17.49	Unknown	0.1	
17.69	Siloxane	1.6	
17.79	Alkane	0.1	
17.86	Unknown	0.1	
18.26	Unknown	<0.1	
18.40	Unknown	<0.1	
18.50	Alkane	0.1	
18.73	Alkane	0.2	
19.21	Siloxane	1.1	
20.55	Siloxane	0.1	
21.69	Siloxane	0.1	
22.70	Siloxane	<0.1	
23.58	Siloxane	<0.1	

Table 2: GC/MS Analysis Results for the Heated Exemplar Material SPME Sample

Retention Time, Minutes	Type of Compound	Concentration, micrograms detected per gram of sample	Notes
13.10	Co-elution of an Alkylbenzene and a Second Unidentified Compound	0.4	
13.65	Best Matches are Alkylbenzenes	0.2	
13.83	Alkane	0.1	
14.19	Best Matches are Alkylbenzenes	0.1	
14.84	Best Matches are Alkylbenzenes	0.1	
14.96	Unknown	0.6	
15.48	Alkane	0.3	
15.59	Unknown	0.1	
15.71	Siloxane	0.3	
16.19	Unknown	0.1	
16.66	Polyaromatic Hydrocarbon (PAH)	0.2	
16.72	Alkane	0.4	
16.86	Unknown	0.1	
17.14	Unknown	0.1	
17.40	Unknown	<0.1	
17.48	Alkane	0.1	
17.69	Siloxane	0.7	
17.77	Alkane	0.6	
17.84	Amine	2.1	
17.88	Polyaromatic Hydrocarbon (PAH)	0.1	

Table 2: GC/MS Analysis Results for the Heated Exemplar Material SPME Sample (Continued)

Retention Time, Minutes	Type of Compound	Concentration, micrograms detected per gram of sample	Notes
18.04	Polyaromatic Hydrocarbon (PAH)	0.1	
18.25	Unknown	0.1	
18.33	Unknown	<0.1	
18.39	Alkane	0.1	
18.45	Alkane	<0.1	
18.49	Alkane	<0.1	
18.54	Unknown	<0.1	
18.72	Alkane	0.5	
19.21	Siloxane	0.6	
19.35	Unknown	0.1	
19.60	Alkane	0.3	
20.42	Alkane	0.2	
20.54	Siloxane	0.1	
20.78	Alkane	0.1	
21.22	Alkane	0.2	
21.68	Siloxane	<0.1	

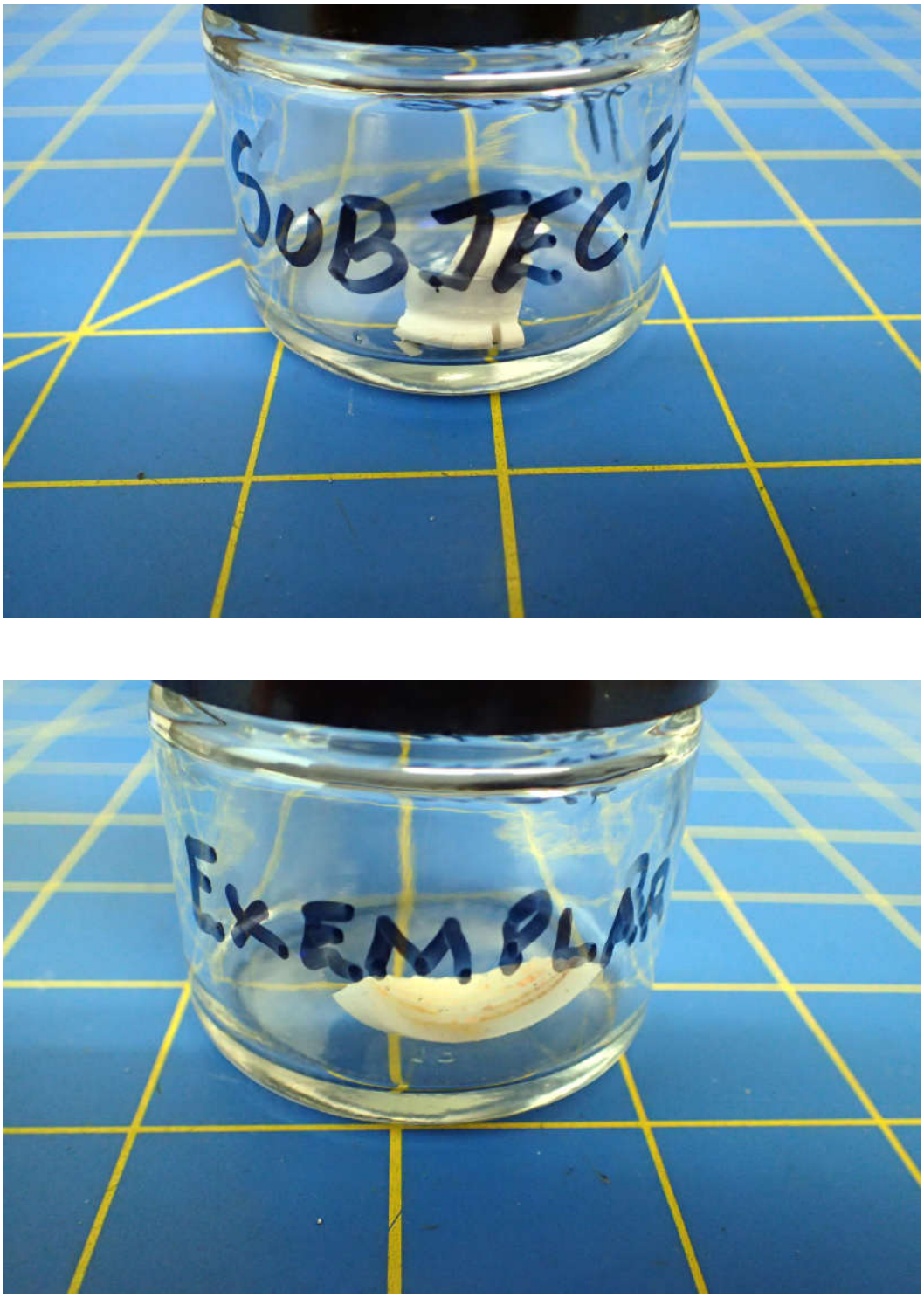


Figure 1:
Photographs showing the subject (top) and exemplar (bottom) samples received for analysis, on a one-inch grid.

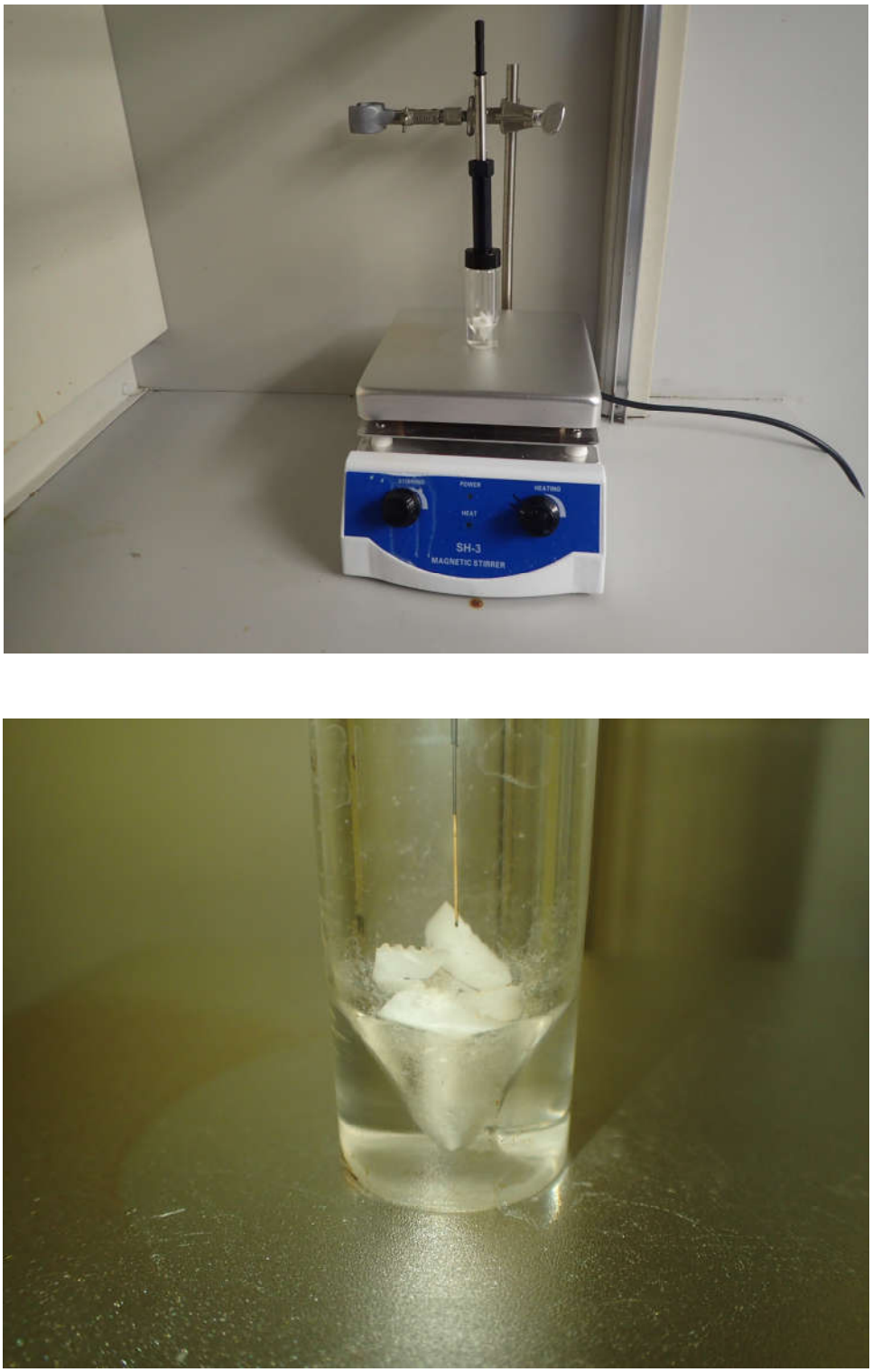


Figure 2:
Photographs showing the SPME analysis setup (top) and a magnified view of the sample and the SPME fiber in the sampling vial (bottom).

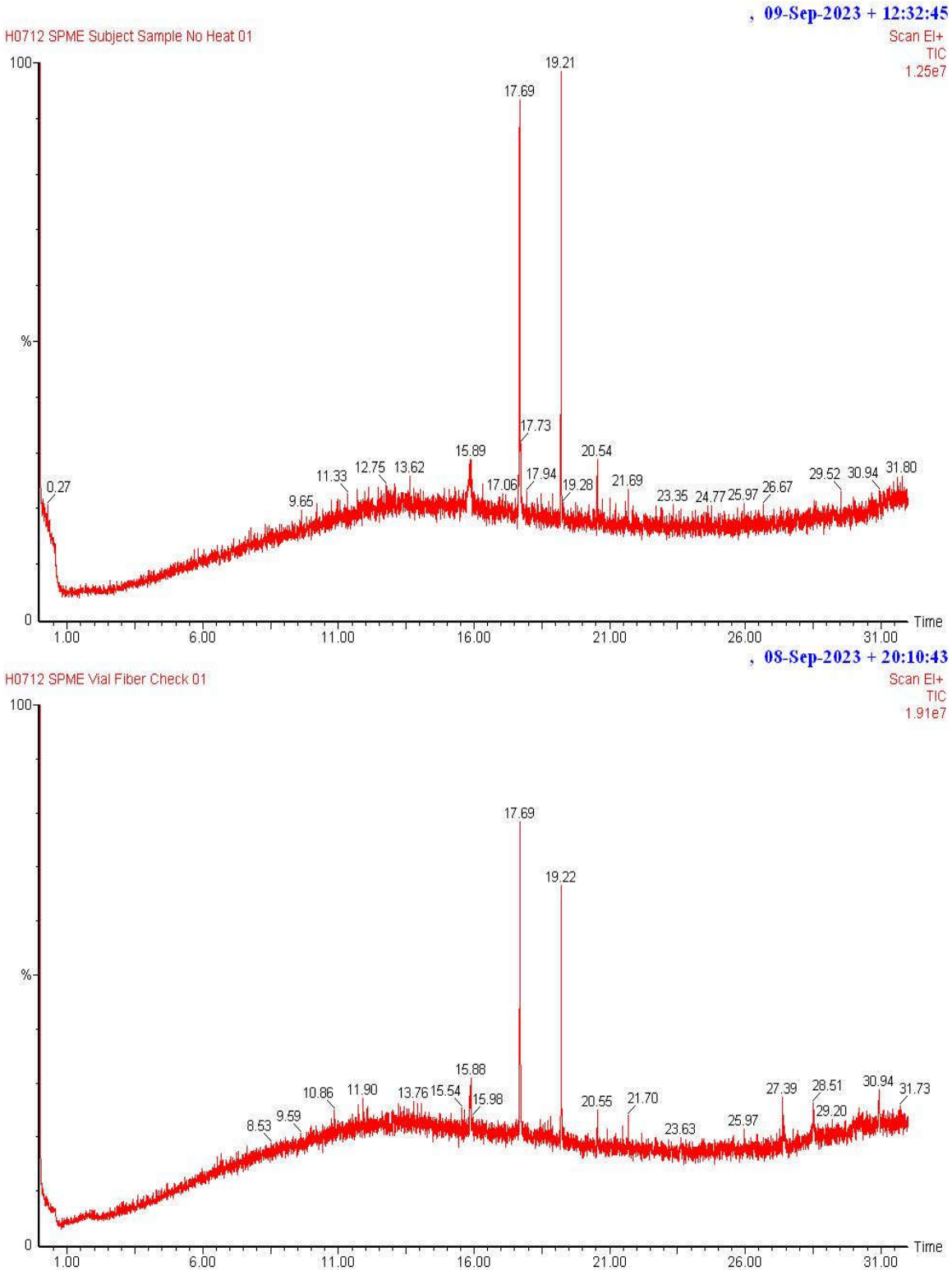


Figure 3:
Total ion chromatograms of the subject sample SPME analysis with no heat applied (top) and the SPME vial and fiber check just prior to the introduction of the sample (bottom).

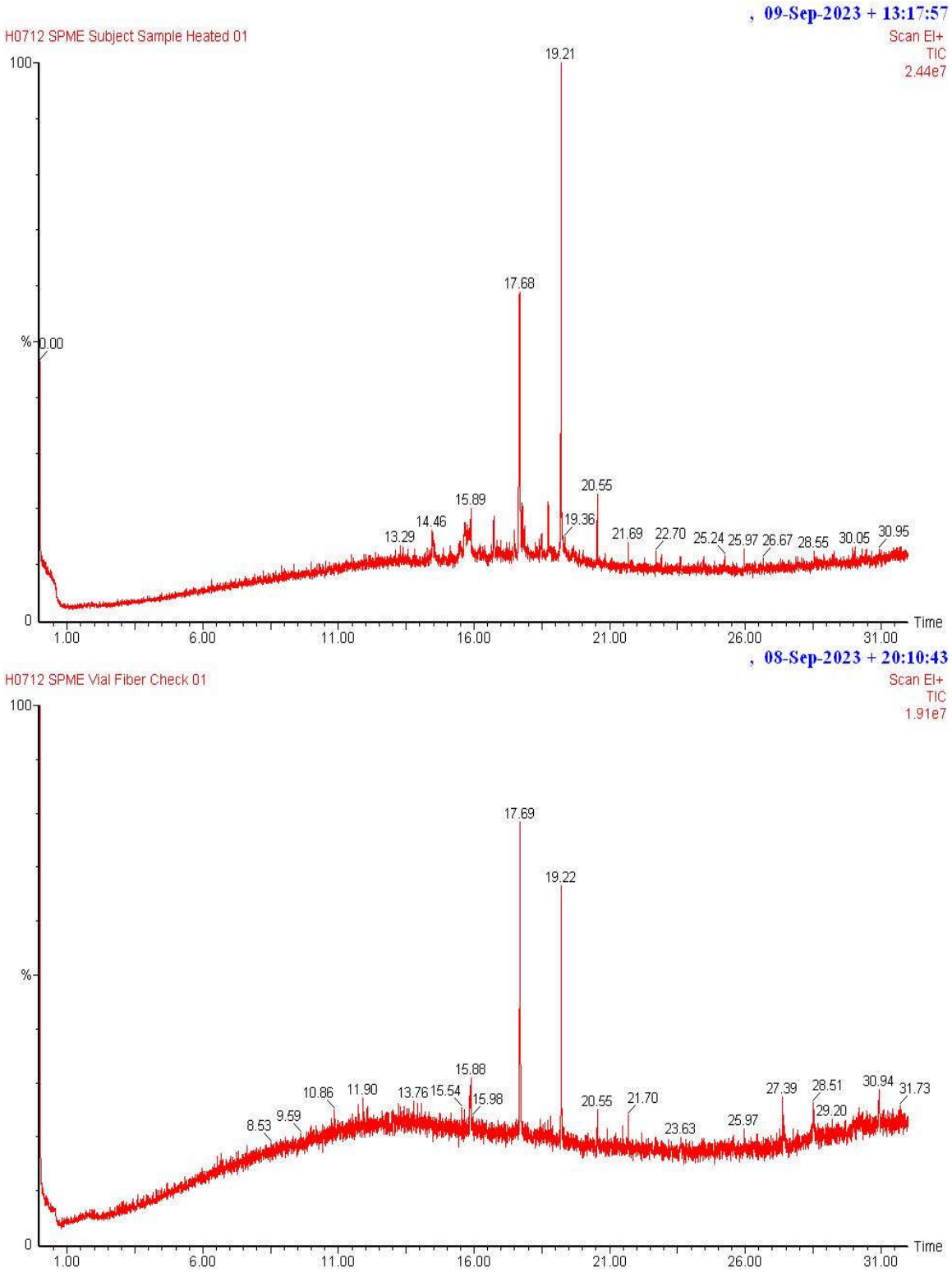


Figure 4:
Total ion chromatograms of the subject sample SPME analysis with heat applied (top) and the SPME vial and fiber check just prior to the introduction of the sample (bottom).

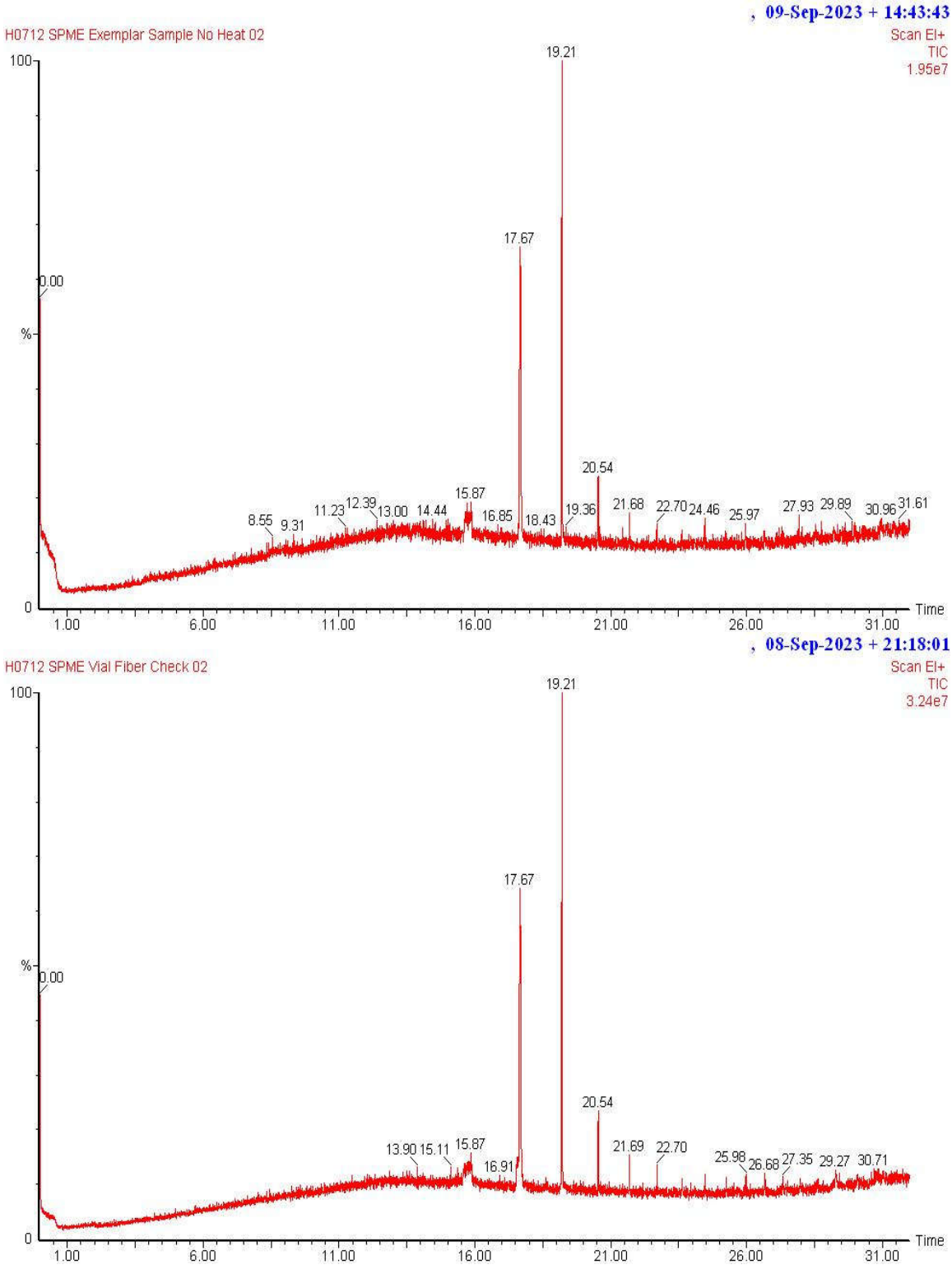


Figure 5:
Total ion chromatograms of the exemplar sample SPME analysis with no heat applied (top) and the SPME vial and fiber check just prior to the introduction of the sample (bottom).

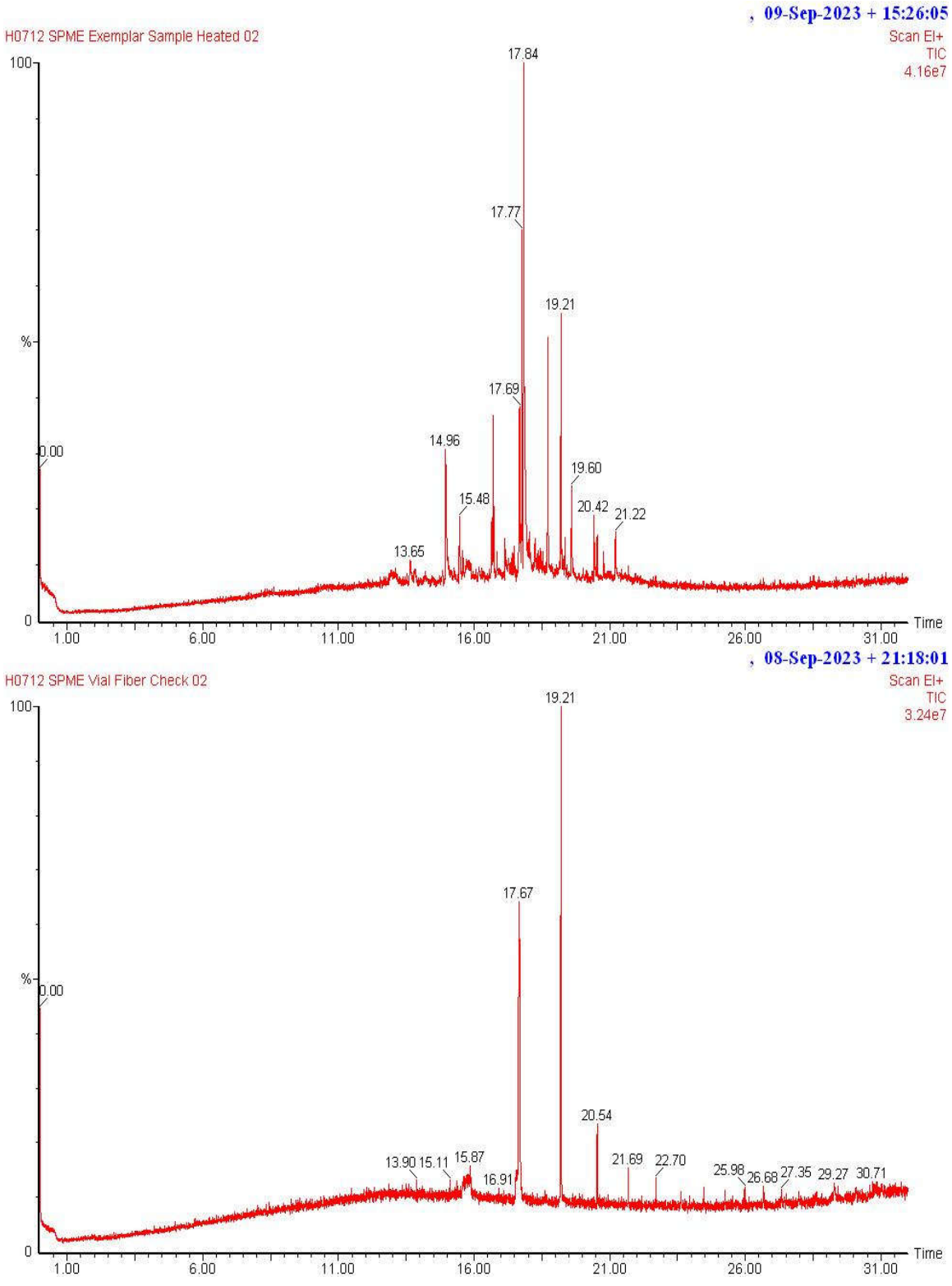


Figure 6:
Total ion chromatograms of the exemplar sample SPME analysis with heat applied (top) and the SPME vial and fiber check just prior to the introduction of the sample (bottom).

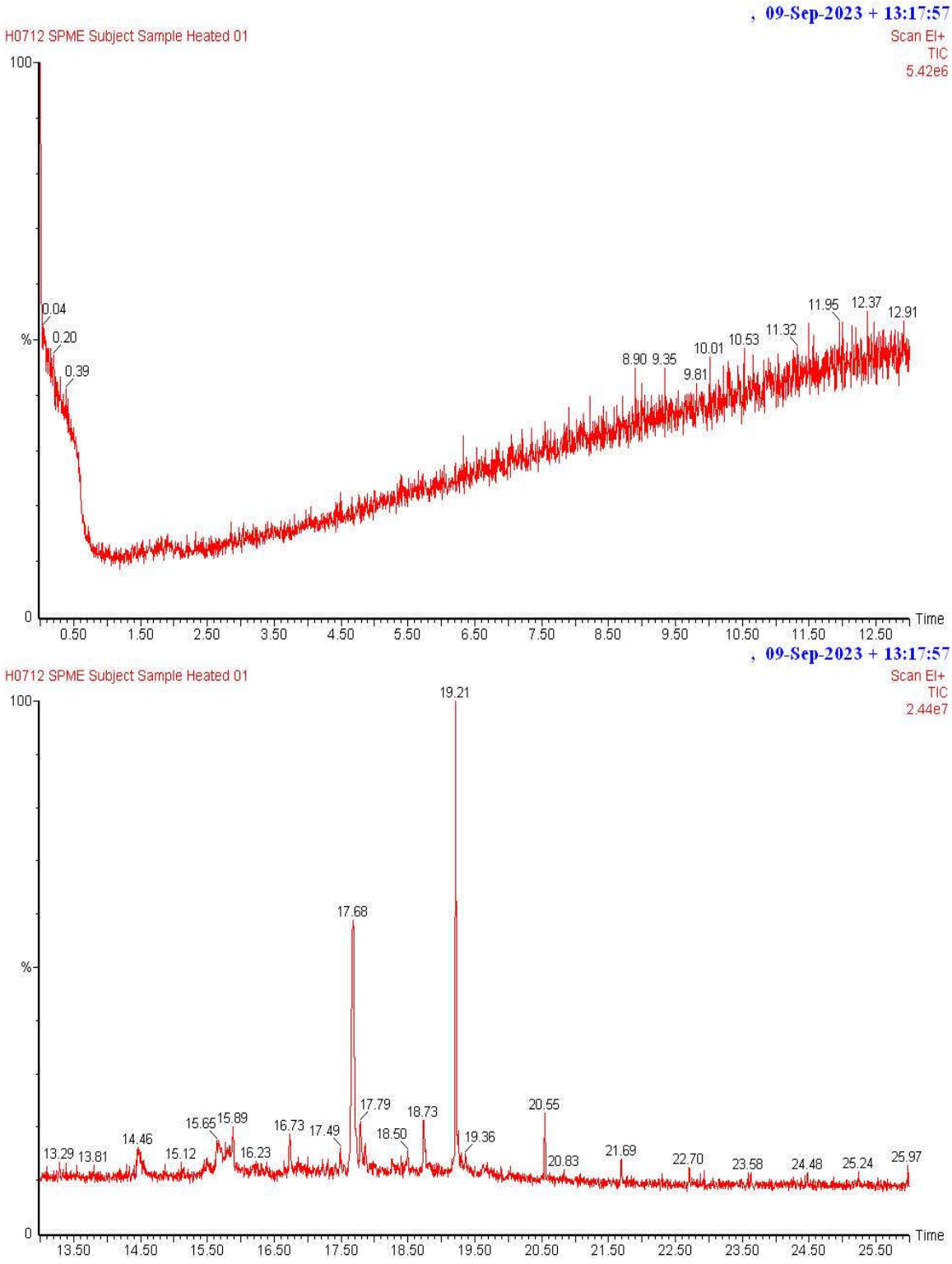


Figure 7:
Expanded baseline views of the total ion chromatogram of the subject sample SPME analysis with heat applied from the injection start to 13 minutes (top) and from 13 minutes to 26 minutes (bottom).

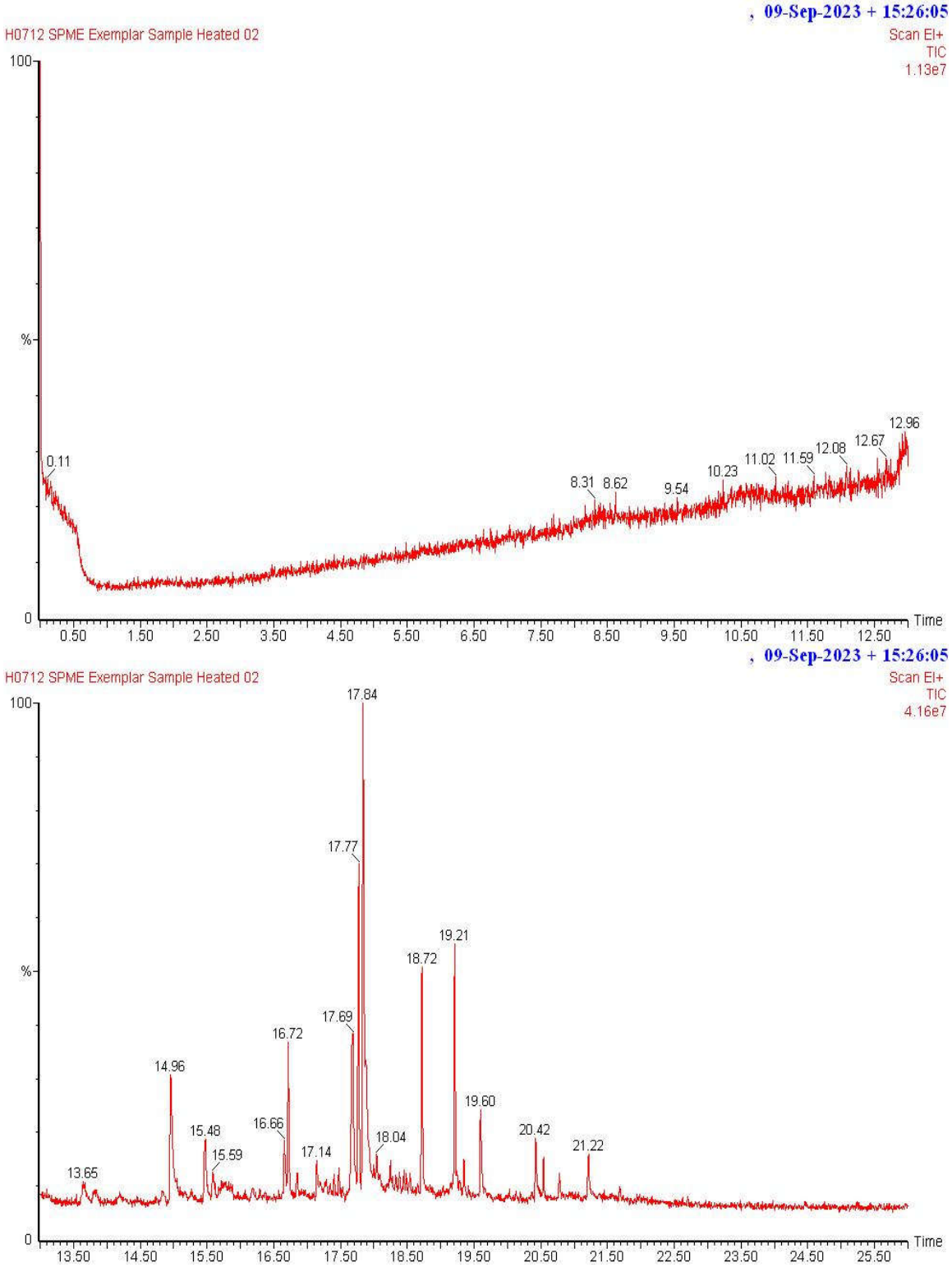


Figure 8:
Expanded baseline views of the total ion chromatogram of the exemplar sample SPME analysis with heat applied from the injection start to 13 minutes (top) and from 13 minutes to 26 minutes (bottom).

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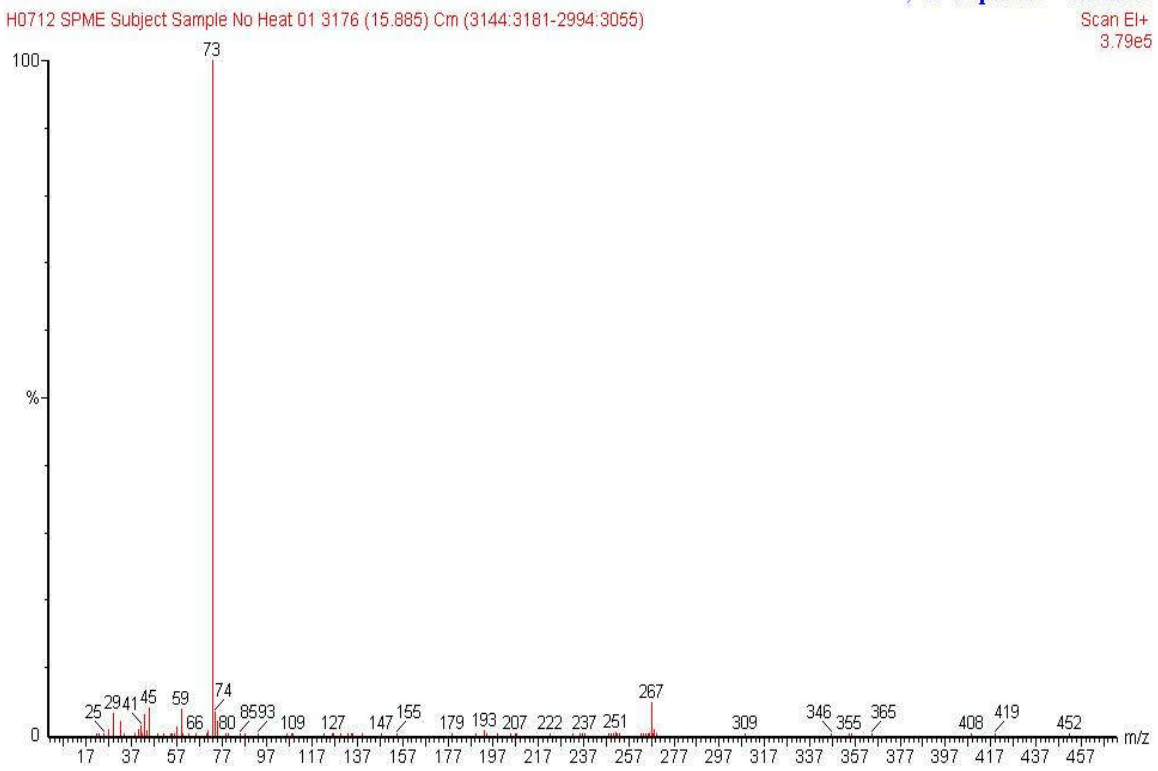
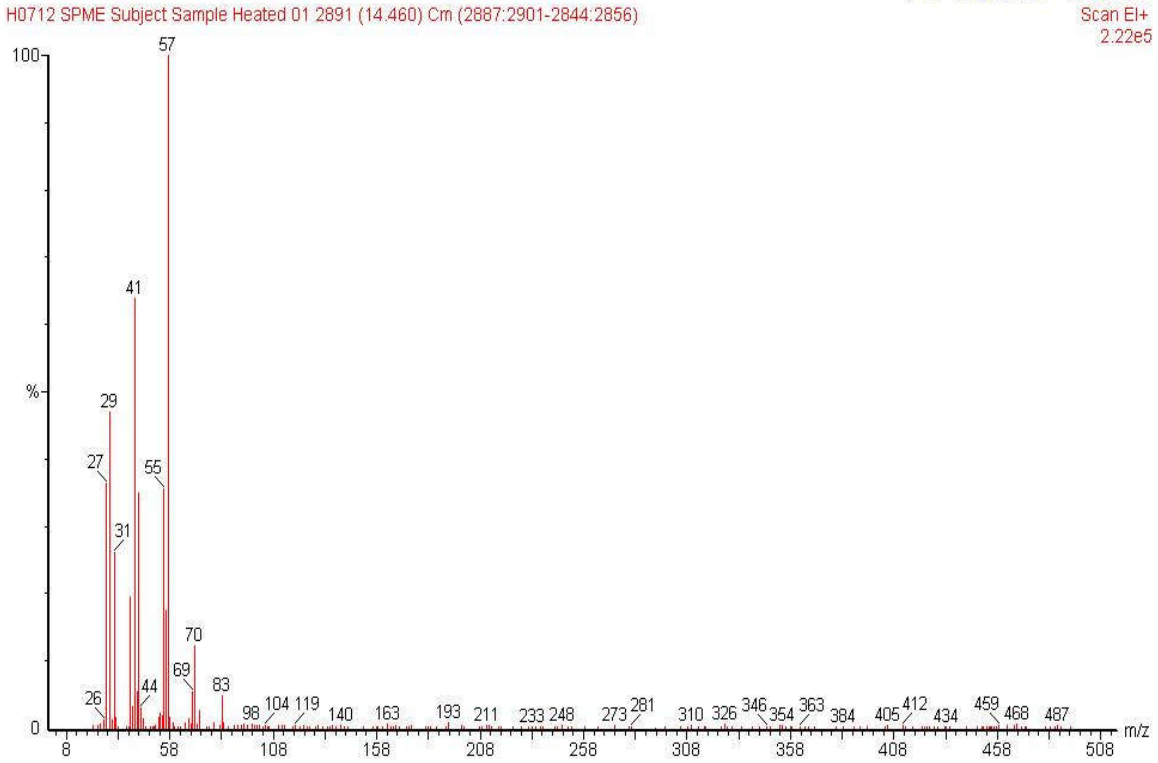


Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	789	718	SILANE, TETRAMETHYL-	88	C4H12Si	75-76-3
2	784	710	SILANE, TRIMETHYL-3-PENTEN-2-YL-, TRANS	142	C8H18Si	53264-56-5
3	752	695	SILANE, TRIMETHYL-2-PENTENYL-, (E)-	142	C8H18Si	40795-28-6
4	786	686	CYCLOPROPANE, 2-METHYLENE-1-PENTYL-1-TRIMETHYLSILYL-	196	C12H24Si	167300-47-2
5	763	686	SILANE, TRIMETHYL-2-PROPENYL-	114	C6H14Si	762-72-1
6	740	674	SILANE, 2-HEXENYLTRIMETHYL-	156	C9H20Si	17898-20-3
7	765	670	SILANE, TRIMETHYL(1-METHYL-1-PROPENYL)-, (E)-	128	C7H16Si	10111-13-4
8	737	660	SILANE, TRIMETHYL[2-METHYLENE-1-(4-PENTENYL)CYCLOPROPYL]-	194	C12H22Si	97778-15-9
9	763	658	1,6-OCTADIENE, 3,8-BIS(TRIMETHYLSILYL)-	254	C14H30Si2	900149-30-5
10	761	656	2,6-OCTADIENE, 1,8-BIS(TRIMETHYLSILYL)-	254	C14H30Si2	900149-32-8
11	749	654	SILANE, (1-CYCLOPENTEN-1-YLMETHYL)TRIMETHYL-	154	C9H18Si	75311-60-3
12	727	654	SILANE, 2-BUTENYLTRIMETHYL-, (E)-	128	C7H16Si	17486-12-3
13	727	654	SILANE, 2-BUTENYLTRIMETHYL-, (Z)-	128	C7H16Si	17486-13-4
14	757	652	SILANE, TRIMETHYL(3-METHYL-3-PHENYL-1-CYCLOPROPEN-1-YL)-	202	C13H18Si	128889-54-3

Figure 9:
Mass spectra of the subject sample with no heat applied SPME sample analysis peak at 15.89 minutes (top) and a NIST library search list (bottom).



Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	861	847	1-HEXANOL, 2-ETHYL-	130	C8H18O	104-76-7
2	860	846	1-PENTANOL, 2-ETHYL-4-METHYL-	130	C8H18O	106-67-2
3	854	835	OXALIC ACID, ALLYL HEPTYL ESTER	228	C12H20O4	900309-23-5
4	842	819	1-BUTANOL, 2-METHYL-, (+/-)-	88	C5H12O	34713-94-5
5	832	814	OXALIC ACID, HEPTYL PROPYL ESTER	230	C12H22O4	900309-26-0
6	836	813	1-BUTANOL, 2-METHYL-, (S)-	88	C5H12O	1565-80-6
7	832	804	1-BUTANOL, 2-METHYL-	88	C5H12O	137-32-6
8	822	804	2-PROPYL-1-PENTANOL	130	C8H18O	58175-57-8
9	805	792	OXALIC ACID, CYCLOBUTYL HEPTYL ESTER	242	C13H22O4	900309-69-8
10	795	782	ISOOCTANE, (ETHENYLOXY)-	156	C10H20O	37769-62-3
11	854	777	VALERALDEHYDE, 4,4-DIMETHYL-2-METHYLENE-	126	C8H14O	5375-28-0
12	785	767	1-BUTOXY-2-ETHYLHEXANE	186	C12H26O	62625-25-6
13	775	763	OXALIC ACID, ISOBUTYL OCTYL ESTER	258	C14H26O4	900309-37-3
14	770	757	1-PENTANOL, 4-METHYL-2-PROPYL-	144	C9H20O	54004-41-0

Figure 10:
Mass spectra of the subject sample with heat applied SPME sample analysis peak at 14.46 minutes (top) and a NIST library search list (bottom).

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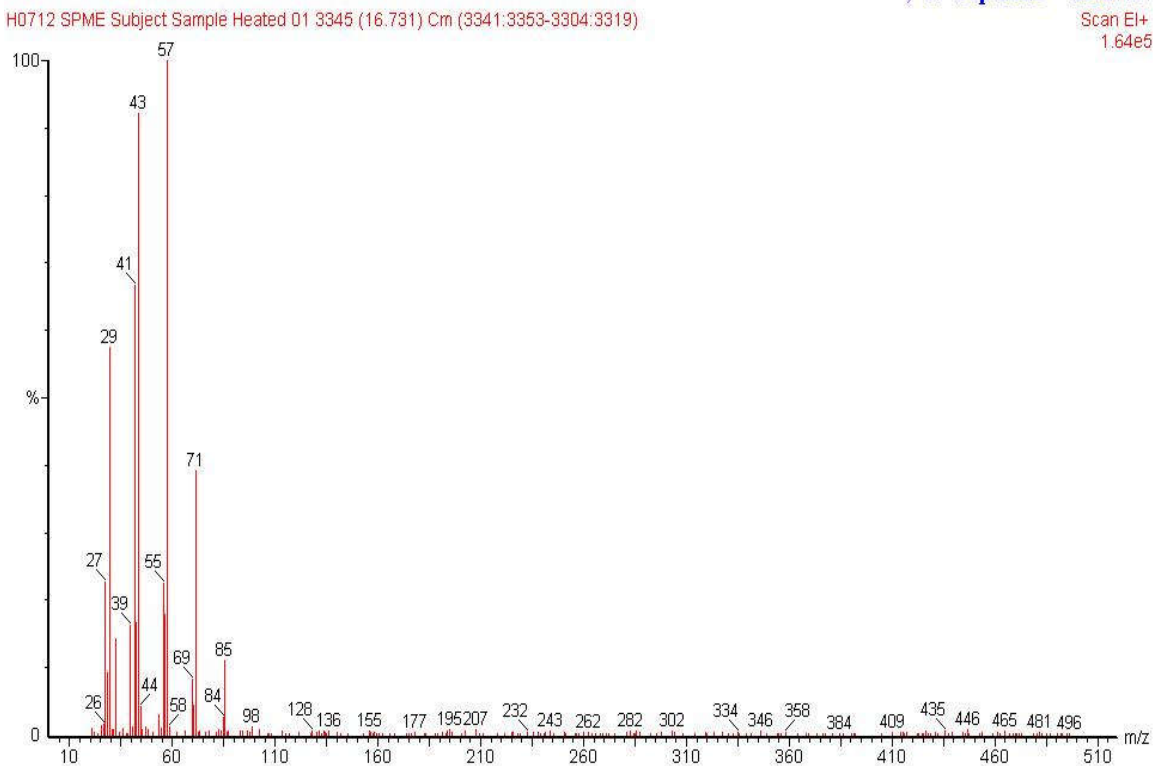

Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	904	864	DECANE	142	C10H22	124-18-5
2	891	851	OXALIC ACID, ISOBUTYL NONYL ESTER	272	C15H28O4	900309-37-4
3	883	844	HEPTANE, 2,6-DIMETHYL-	128	C9H20	1072-05-5
4	883	844	OCTANE, 2-METHYL-	128	C9H20	3221-61-2
5	881	842	OCTANE	114	C8H18	111-65-9
6	880	841	NONANE, 2-METHYL-	142	C10H22	871-83-0
7	878	839	2-BROMONONANE	206	C9H19Br	2216-35-5
8	877	838	TRIDECANE	184	C13H28	629-50-5
9	876	837	DECANE, 2-METHYL-	156	C11H24	6975-98-0
10	875	836	PENTANE, 2,2,3,4-TETRAMETHYL-	128	C9H20	1186-53-4
11	873	834	HEXANE, 2,4-DIMETHYL-	114	C8H18	589-43-5
12	871	832	NONANE	128	C9H20	111-84-2
13	867	829	OCTANE, 2,7-DIMETHYL-	142	C10H22	1072-16-8
14	863	825	HEXANE, 2,3,4-TRIMETHYL-	128	C9H20	921-47-1

Figure 11:
Mass spectra of the subject sample with heat applied SPME sample analysis peak at 16.73 minutes (top) and a NIST library search list (bottom).

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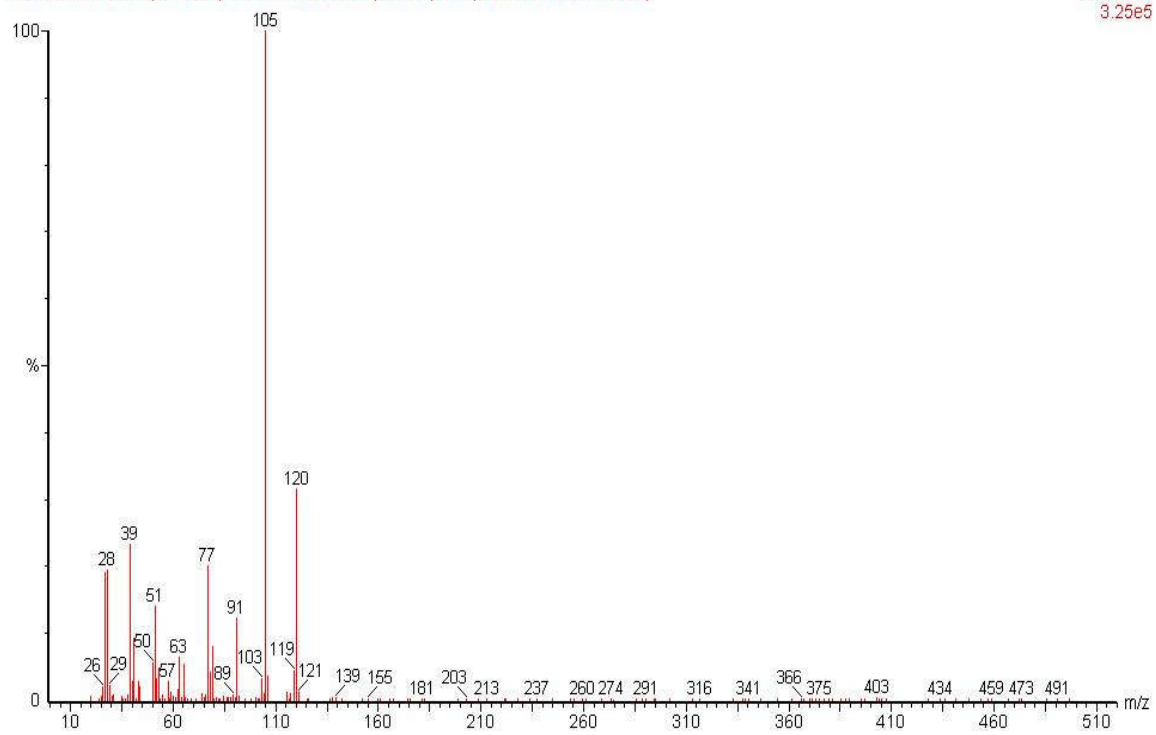
H0712 SPME Exemplar Sample Heated 02 2729 (13.650) Cm (2722:2738-2656:2674)

Scan EI+
3.25e5



Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	890	864	BENZENE, 1,2,4-TRIMETHYL-	120	C9H12	95-63-6
2	862	826	BENZENE, 1,2,3-TRIMETHYL-	120	C9H12	526-73-8
3	868	786	1,3,5-CYCLOHEPTATRIENE, 7,7-DIMETHYL-	120	C9H12	7557-11-1
4	862	784	BENZENE, 1-ETHYL-3-METHYL-	120	C9H12	620-14-4
5	861	782	BENZENE, 1-ETHYL-4-METHYL-	120	C9H12	622-96-8
6	857	778	BENZENE, 1-ETHYL-2-METHYL-	120	C9H12	611-14-3
7	848	770	1-HEPTEN-5-YNE, 2-METHYL-3-METHYLENE-	120	C9H12	76003-40-2
8	845	768	BENZENE, 1,3,5-TRIMETHYL-	120	C9H12	108-67-8
9	857	766	2,4-NONADIYNE	120	C9H12	63621-15-8
10	852	763	BENZENE, (1-METHYLETHYL)-	120	C9H12	98-82-8
11	862	733	2,3-HEPTADIEN-5-YNE, 2,4-DIMETHYL-	120	C9H12	41898-89-9
12	807	729	CYCLOHEXANE, 1,2,4-TRIS(METHYLENE)-	120	C9H12	14296-81-2
13	860	722	BENZENE, (1-METHYL-3-BUTENYL)-	146	C11H14	10340-49-5
14	740	716	BENZENE, (1-METHYLPENTYL)-	162	C12H18	6031-02-3

Figure 12:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 13.65 minutes (top) and a NIST library search list (bottom).

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H0712 SPME Exemplar Sample Heated 02 2838 (14.195) Cm (2829:2848-2807:2825)

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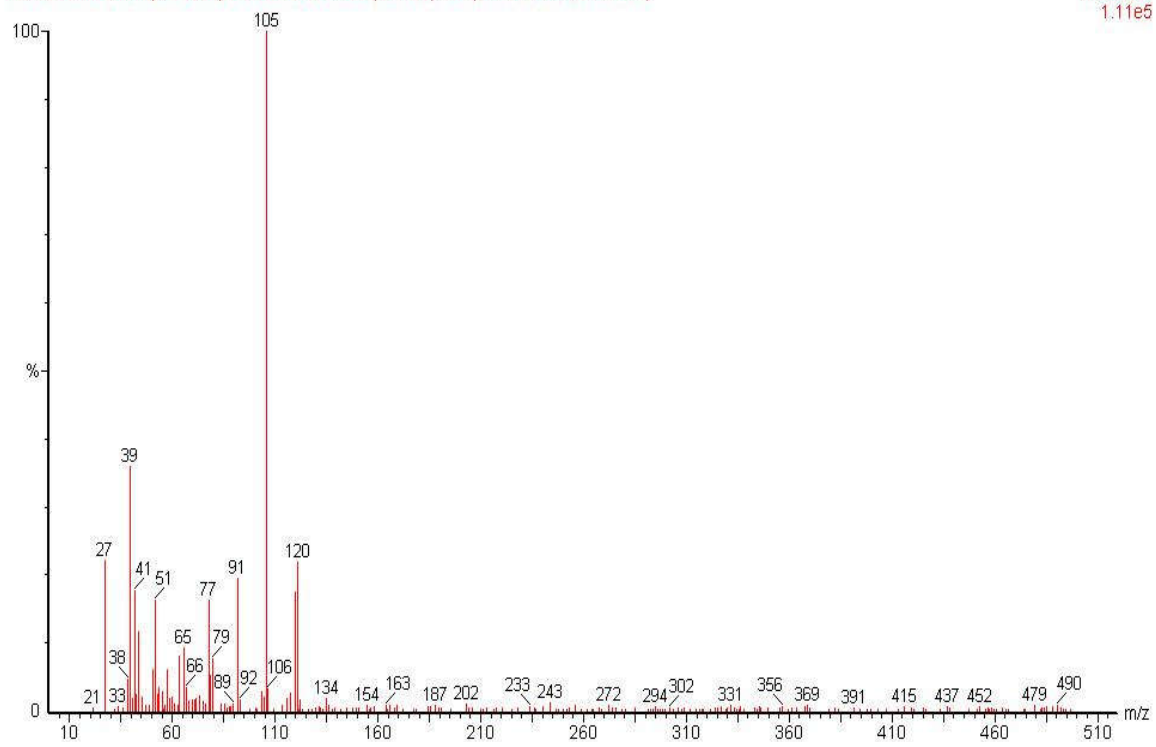
1.11e5



Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	805	737	1-HEPTEN-5-YNE, 2-METHYL-3-METHYLENE-	120	C9H12	76003-40-2
2	799	723	BENZENE, 1,2,3-TRIMETHYL-	120	C9H12	526-73-8
3	794	720	2,4-NONADIYNE	120	C9H12	63621-15-8
4	789	716	1,3,5-CYCLOHEPTATRIENE, 7,7-DIMETHYL-	120	C9H12	7557-11-1
5	781	702	BENZENE, 1-ETHYL-4-METHYL-	120	C9H12	622-96-8
6	753	694	BENZENE, 1,3,5-TRIMETHYL-	120	C9H12	108-67-8
7	755	684	BENZENE, 1,2,4-TRIMETHYL-	120	C9H12	95-63-6
8	736	674	CYCLOHEXANE, 1,2,4-TRIS(METHYLENE)-	120	C9H12	14296-81-2
9	769	654	1,3-CYCLOPENTADIENE, 5-(1-METHYLPROPYLIDENE)-	120	C9H12	3141-02-4
10	765	651	2,3-HEPTADIEN-5-YNE, 2,4-DIMETHYL-	120	C9H12	41898-89-9
11	707	647	1-HEXEN-4-YNE, 3-ETHYLIDENE-2-METHYL-	120	C9H12	76003-39-9
12	770	608	2-BROMOISOBUTYROPHENONE	226	C10H11OBr	10409-54-8
13	660	604	CYCLONONA-1,2,6-TRIENE	120	C9H12	900196-99-9
14	734	597	BENZOIC ACID, 4-METHYL-, 2-OXO-2-PHENYLETHYL ESTER	254	C16H14O3	54797-44-3

Figure 13:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 14.19 minutes (top) and a NIST library search list (bottom).

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H0712 SPME Exemplar Sample Heated 02 2990 (14.955) Cm (2988:2994-(2976:2982+3069:3076))

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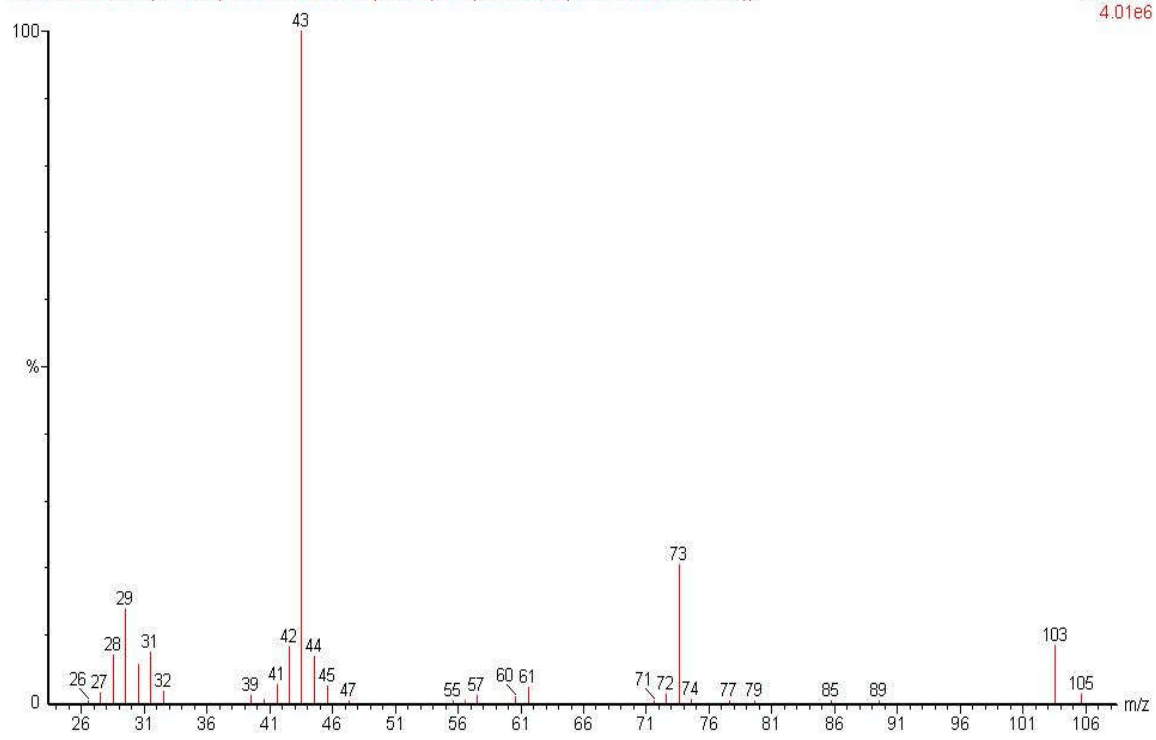
4.01e6



Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	880	809	ACETIC ACID, (ACETYLOXY)-	118	C4H6O4	13831-30-6
2	805	793	METHANE, DIPROPOXY-	132	C7H16O2	505-84-0
3	845	780	1,4-DIOXANE-2,3-DIOL, DIACETATE	204	C8H12O6	15874-26-7
4	828	749	1,2-EPOXY-3-PROPYL ACETATE	116	C5H8O3	6387-99-9
5	734	721	PROPANE-1,1-DIOL DIACETATE	160	C7H12O4	33931-80-5
6	763	718	2-PROPANONE, 1-(1,3-DIOXOLAN-2-YL)-	130	C6H10O3	767-04-4
7	793	711	SULFUROUS ACID, DIPROPYL ESTER	166	C6H14O3S	623-98-3
8	767	709	1,2,3-PROPANETRIOL, 1-ACETATE	134	C5H10O4	106-61-6
9	715	702	ETHYL N-HYDROXYACETIMIDATE	103	C4H9O2N	10576-12-2
10	756	699	2-PROPANONE, 1-HYDROXY-	74	C3H6O2	116-09-6
11	743	698	ETHYL ACETATE	88	C4H8O2	141-78-6
12	734	688	METHANOL, (METHYL-ONN-AZOXY)-, ACETATE (ESTER)	132	C4H8O3N2	592-62-1
13	710	686	1,2-ETHANEDIOL, DIACETATE	146	C6H10O4	111-55-7
14	735	681	ETHANOL, 2-CHLORO-, ACETATE	122	C4H7O2Cl	542-58-5

Figure 14:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 14.96 minutes (top) and a NIST library search list (bottom).

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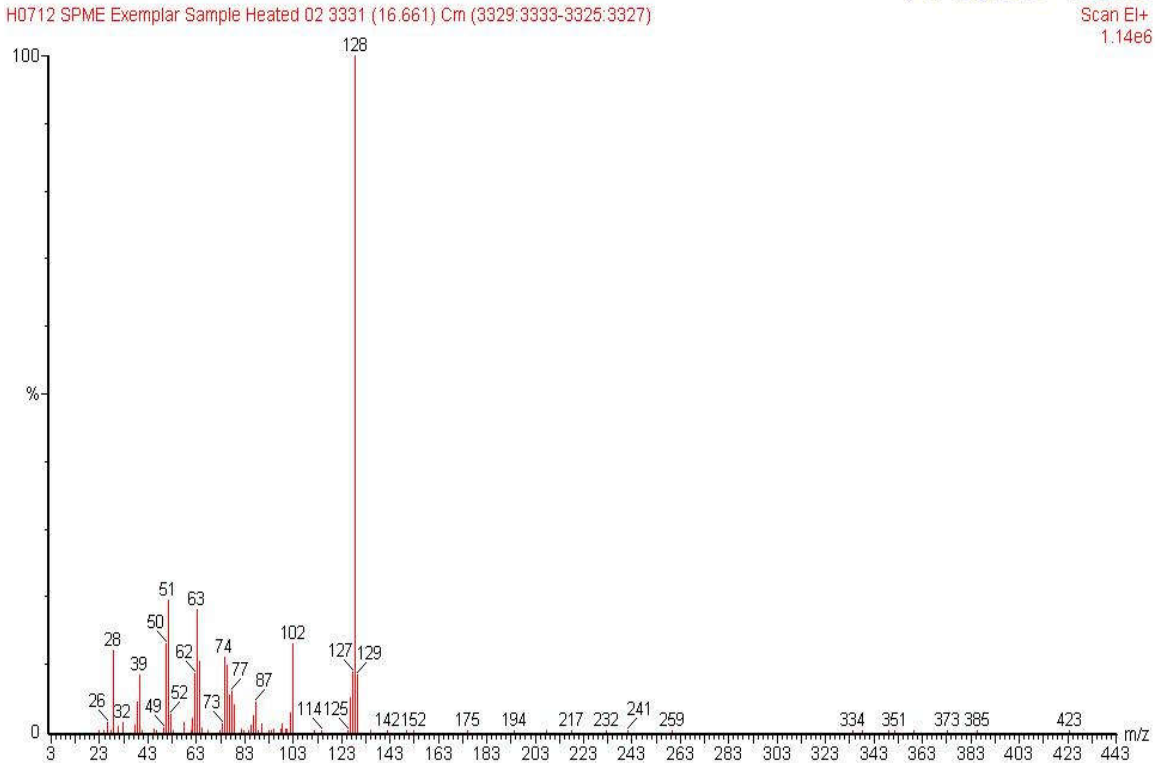

Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	967	872	1H-INDENE, 1-METHYLENE-	128	C10H8	2471-84-3
2	946	849	4-PHENYLBUT-3-ENE-1-YNE	128	C10H8	900222-12-0
3	896	848	AZULENE	128	C10H8	275-51-4
4	891	829	NAPHTHALENE	128	C10H8	91-20-3
5	894	829	[4.2.2]PROPELLA-2,4,7,9-TETRAENE	128	C10H8	88090-34-0
6	761	716	ACETIC ACID, 2-(2-HYDROXY-1-NAPHTHYLMETHYLAMINO)-	231	C13H13O3N	87338-88-3
7	778	714	1,3-BENZENEDICARBONITRILE	128	C8H4N2	626-17-5
8	769	713	1,4-BENZENEDICARBONITRILE	128	C8H4N2	623-26-7
9	768	712	1,2-BENZENEDICARBONITRILE	128	C8H4N2	91-15-6
10	772	710	QUINOLINE, 2-IODO-	255	C9H6NI	6560-83-4
11	734	681	3,4-DIHYDRO-1,2-NAPHTHALENEDICARBOXYLIC ANHYDRIDE	200	C12H8O3	37845-14-0
12	761	672	N-METHYL-9-AZA-TRICYCLO[6.2.2.0(2,7)]DODEC-2,4,6,11-TETRAENE-10-ONE	185	C12H11ON	13131-19-6
13	684	624	CYCLOPROP[A]INDENE, 6-BROMO-1,1A,6,6A-TETRAHYDRO-	208	C10H9Br	55780-41-1
14	678	620	QUINOLINE, 2-NITRO-	174	C9H6O2N2	18714-34-6

Figure 15:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 16.66 minutes (top) and a NIST library search list (bottom).

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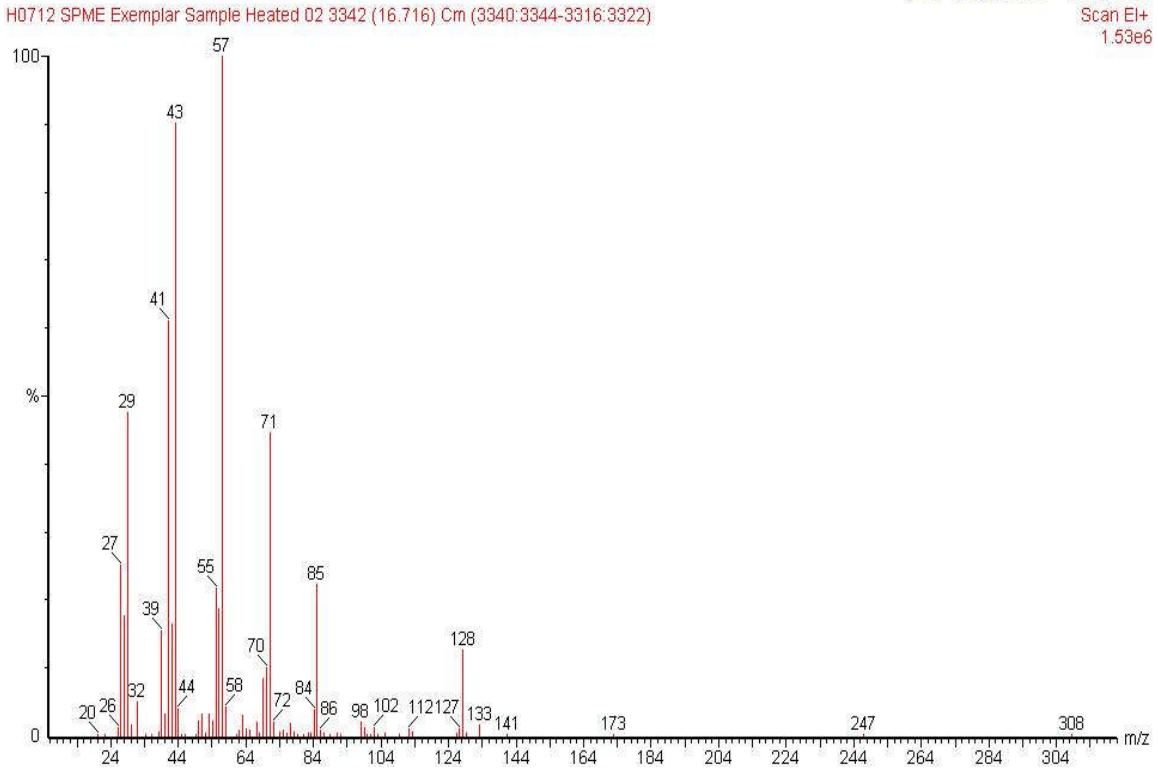
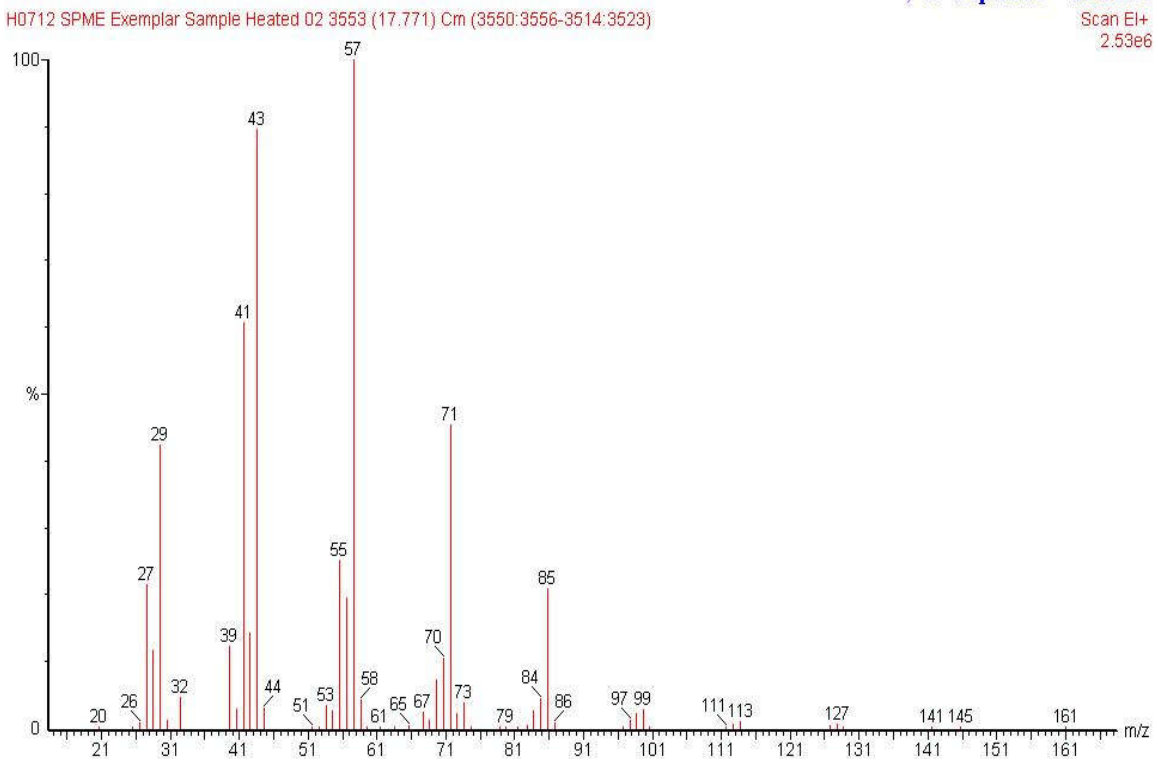


Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	952	900	DECANE	142	C10H22	124-18-5
2	929	899	NONANE	128	C9H20	111-84-2
3	928	897	NONANE, 2-METHYL-	142	C10H22	871-83-0
4	921	890	2-BROMONONANE	206	C9H19Br	2216-35-5
5	918	889	3-HEXANONE, 2,4-DIMETHYL-	128	C8H16O	18641-70-8
6	925	886	3-HEXANONE, 2,2-DIMETHYL-	128	C8H16O	5405-79-8
7	911	884	HEPTANE, 2,6-DIMETHYL-	128	C9H20	1072-05-5
8	915	883	OCTANE, 2-METHYL-	128	C9H20	3221-61-2
9	915	881	4-HEPTANONE, 3-METHYL-	128	C8H16O	15726-15-5
10	934	880	OXALIC ACID, ISOBUTYL NONYL ESTER	272	C15H28O4	900309-37-4
11	924	879	HEXANE, 2,4-DIMETHYL-	114	C8H18	589-43-5
12	916	875	TETRADECANE, 1-IODO-	324	C14H29I	19218-94-1
13	933	874	TRIDECANE	184	C13H28	629-50-5
14	900	874	HEXANE, 2,3,4-TRIMETHYL-	128	C9H20	921-47-1

Figure 16:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 16.72 minutes (top) and a NIST library search list (bottom).



Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	965	944	DECANE	142	C10H22	124-18-5
2	961	937	OXALIC ACID, ISOBUTYL NONYL ESTER	272	C15H28O4	900309-37-4
3	959	932	TRIDECANE	184	C13H28	629-50-5
4	945	924	NONANE, 2-METHYL-	142	C10H22	871-83-0
5	942	921	DECANE, 2-METHYL-	156	C11H24	6975-98-0
6	933	913	NONANE	128	C9H20	111-84-2
7	938	910	UNDECANE, 3,7-DIMETHYL-	184	C13H28	17301-29-0
8	937	907	DODECANE, 2-METHYL-	184	C13H28	1560-97-0
9	934	906	UNDECANE, 5-ETHYL-	184	C13H28	17453-94-0
10	943	905	OCTANE, 2,4,6-TRIMETHYL-	156	C11H24	62016-37-9
11	933	905	2-BROMONONANE	206	C9H19Br	2216-35-5
12	942	902	UNDECANE	156	C11H24	1120-21-4
13	921	901	DECANE, 2,4-DIMETHYL-	170	C12H26	2801-84-5
14	944	900	DECANE, 2,9-DIMETHYL-	170	C12H26	1002-17-1

Figure 17:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 17.77 minutes (top) and a NIST library search list (bottom).

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H0712 SPME Exemplar Sample Heated 02 3566 (17.836) Cm (3563:3572-3627:3634)

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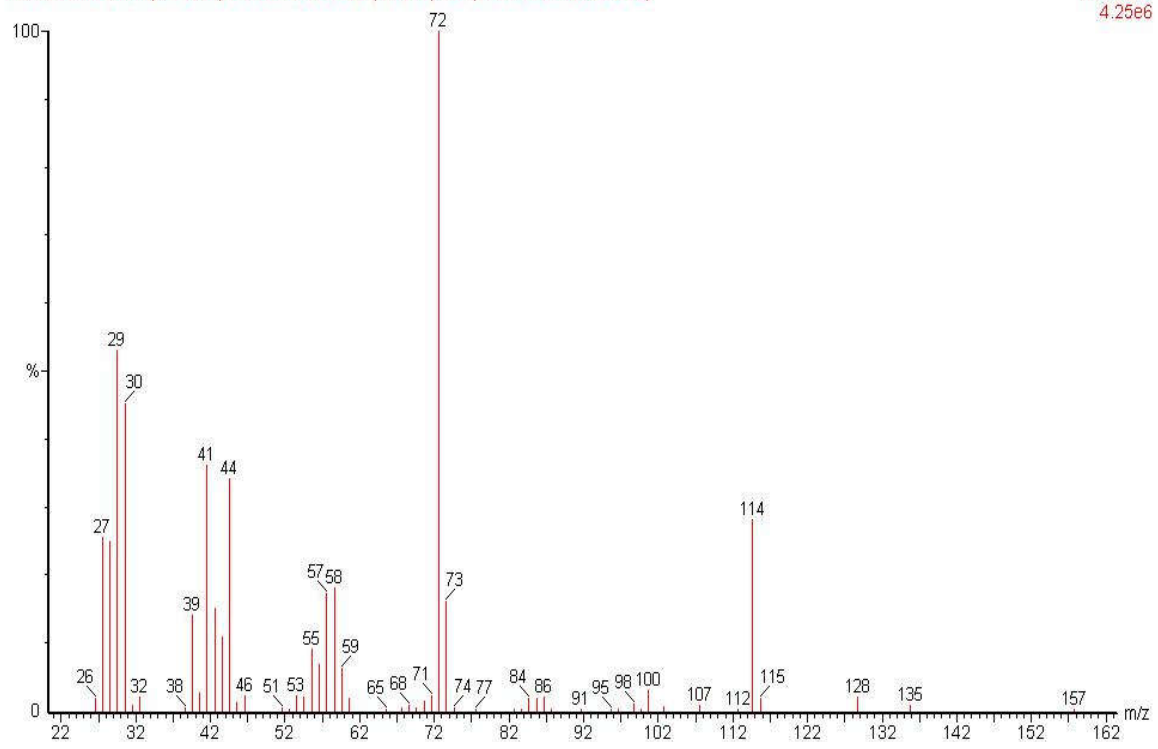
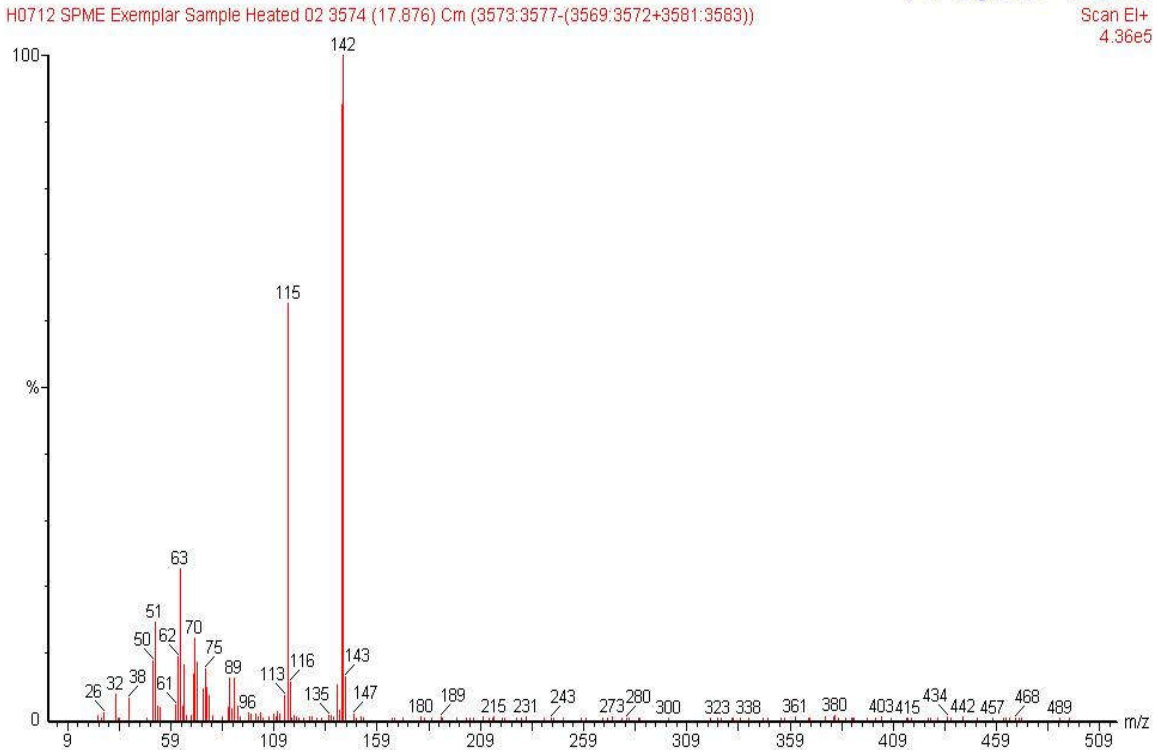


Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	849	809	2-PENTANAMINE, N-ETHYL-4-METHYL-	129	C8H19N	42966-64-3
2	822	798	FORMAMIDE, N,N-DIBUTYL-	157	C9H19ON	761-65-9
3	796	749	1-BUTANAMINE, N-PROPYL-	115	C7H17N	20193-21-9
4	765	749	1-BUTANAMINE, N-(1-METHYLETHYL)-	115	C7H17N	39099-23-5
5	771	737	4-AMINOHEPTANE	115	C7H17N	16751-59-0
6	798	727	PROPYLNEOPENTYLAMINE	129	C8H19N	131229-65-7
7	792	721	2-PROPANAMINE, N-ETHYL-	87	C5H13N	19961-27-4
8	756	720	3-BUTEN-1-AMINE, N-ETHYL-N-METHYL-	113	C7H15N	61308-10-9
9	767	716	1-BUTANAMINE, N-ETHYL-N-METHYL-	115	C7H17N	66225-40-9
10	837	714	1-HEXANAMINE, N-PROPYL-	143	C9H21N	20193-23-1
11	785	712	1,2-ETHANEDIAMINE, N-PROPYL-	102	C5H14N2	111-39-7
12	740	708	ACETAMIDE, N,N-DIPROPYL-	143	C8H17ON	1116-24-1
13	772	691	1-PROPANAMINE, N-ETHYL-N-METHYL-	101	C6H15N	4458-32-6
14	748	688	DL-2-AMINO-1-PENTANOL	103	C5H13ON	4146-04-7

Figure 18:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 17.84 minutes (top) and a NIST library search list (bottom).



Hit	rev	FOR	Compound Name	M.W.	Formula	CAS
1	903	882	BICYCLO[4.4.1]UNDECA-1,3,5,7,9-PENTAENE	142	C11H10	2443-46-1
2	902	876	BENZOCYCLOHEPTATRIENE	142	C11H10	264-09-5
3	895	875	1,4-METHANONAPHTHALENE, 1,4-DIHYDRO-	142	C11H10	4453-90-1
4	894	873	1H-INDENE, 1-ETHYLIDENE-	142	C11H10	2471-83-2
5	894	870	NAPHTHALENE, 1-METHYL-	142	C11H10	90-12-0
6	841	811	NAPHTHALENE, 2-METHYL-	142	C11H10	91-57-6
7	763	683	BENZENEACETONITRILE, 4-CYANO-	142	C9H6N2	876-31-3
8	761	678	BENZENEACETONITRILE, 2-CYANO-	142	C9H6N2	3759-28-2
9	707	661	NAPHTHALENE, 2-(BROMOMETHYL)-	220	C11H9Br	939-26-4
10	689	650	2-NAPHTHYLACETYL CHLOTIDE	204	C12H9OCl	37859-25-9
11	700	646	1-NAPHTHALENEACETAMIDE	185	C12H11ON	86-86-2
12	699	641	1-NAPHTHALENEMETHYL ISOTHIOCYANATE	199	C12H9NS	17112-82-2
13	710	635	ACETONITRILE, 2-(3-CYANOPHENYL)-	142	C9H6N2	16532-78-8
14	704	633	2-NAPHTHALENEETHANOL	172	C12H12O	1485-07-0

Figure 19:
Mass spectra of the exemplar sample with heat applied SPME sample analysis peak at 17.88 minutes (top) and a NIST library search list (bottom).

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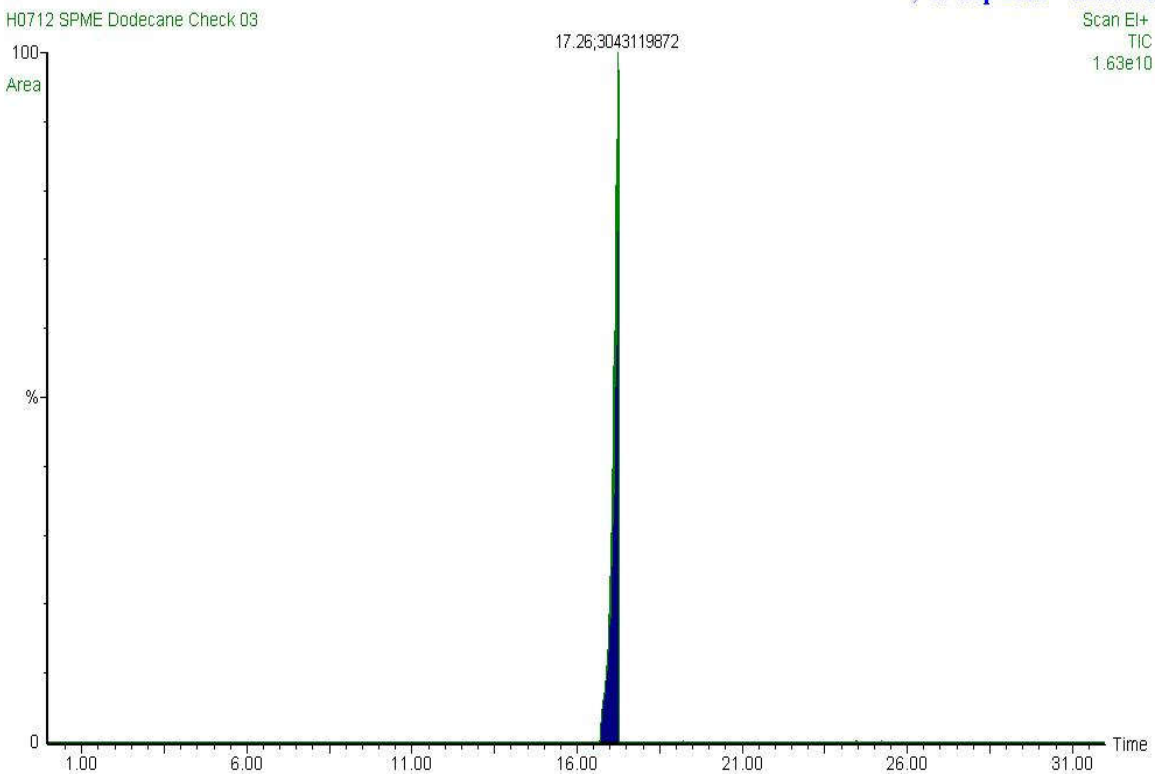

Figure 20:
Total ion chromatogram of the dodecane check standard.

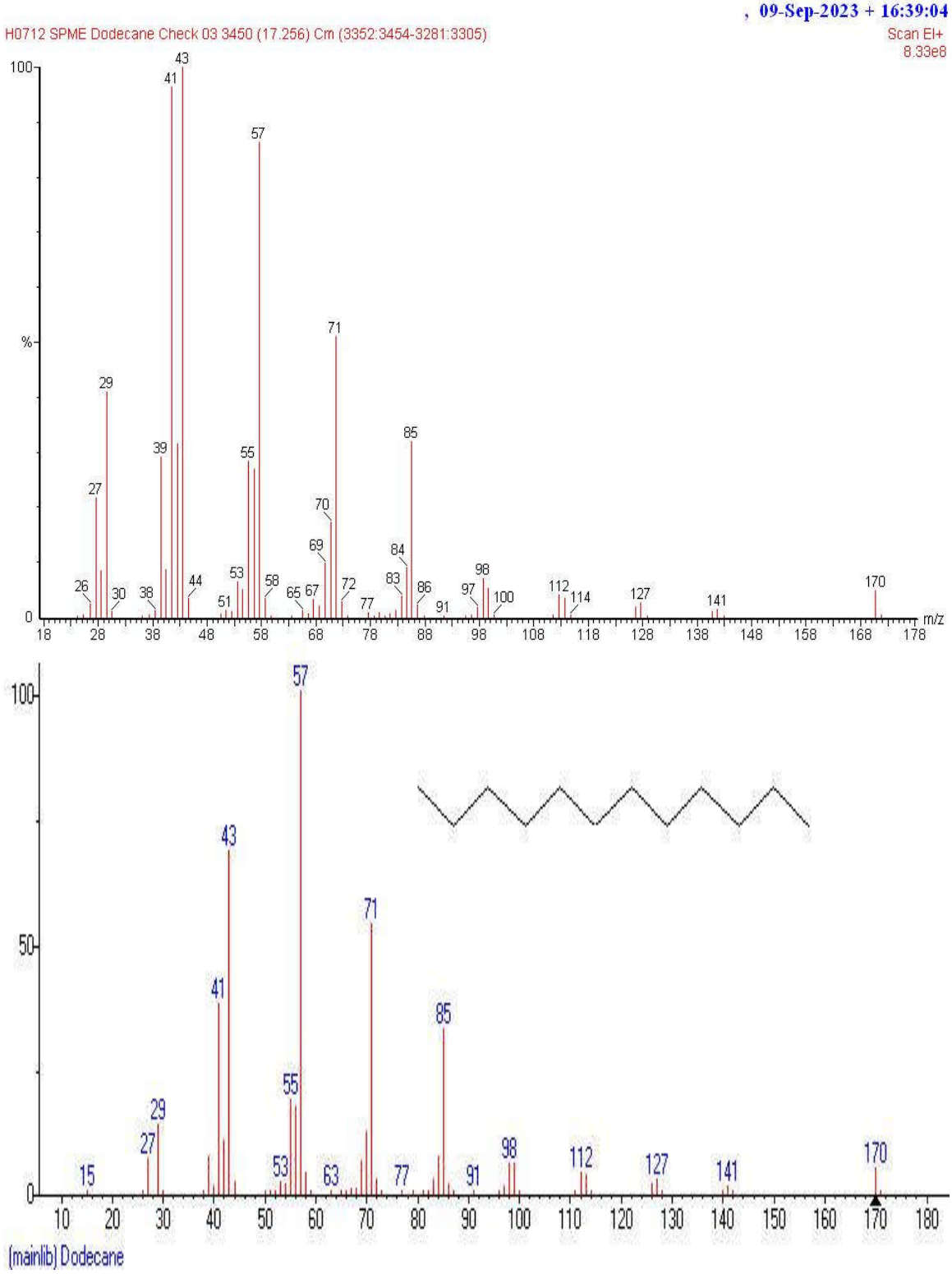

Figure 21:
Mass spectra of the dodecane check standard peak at 17.26 minutes (top) and a NIST library reference (bottom).

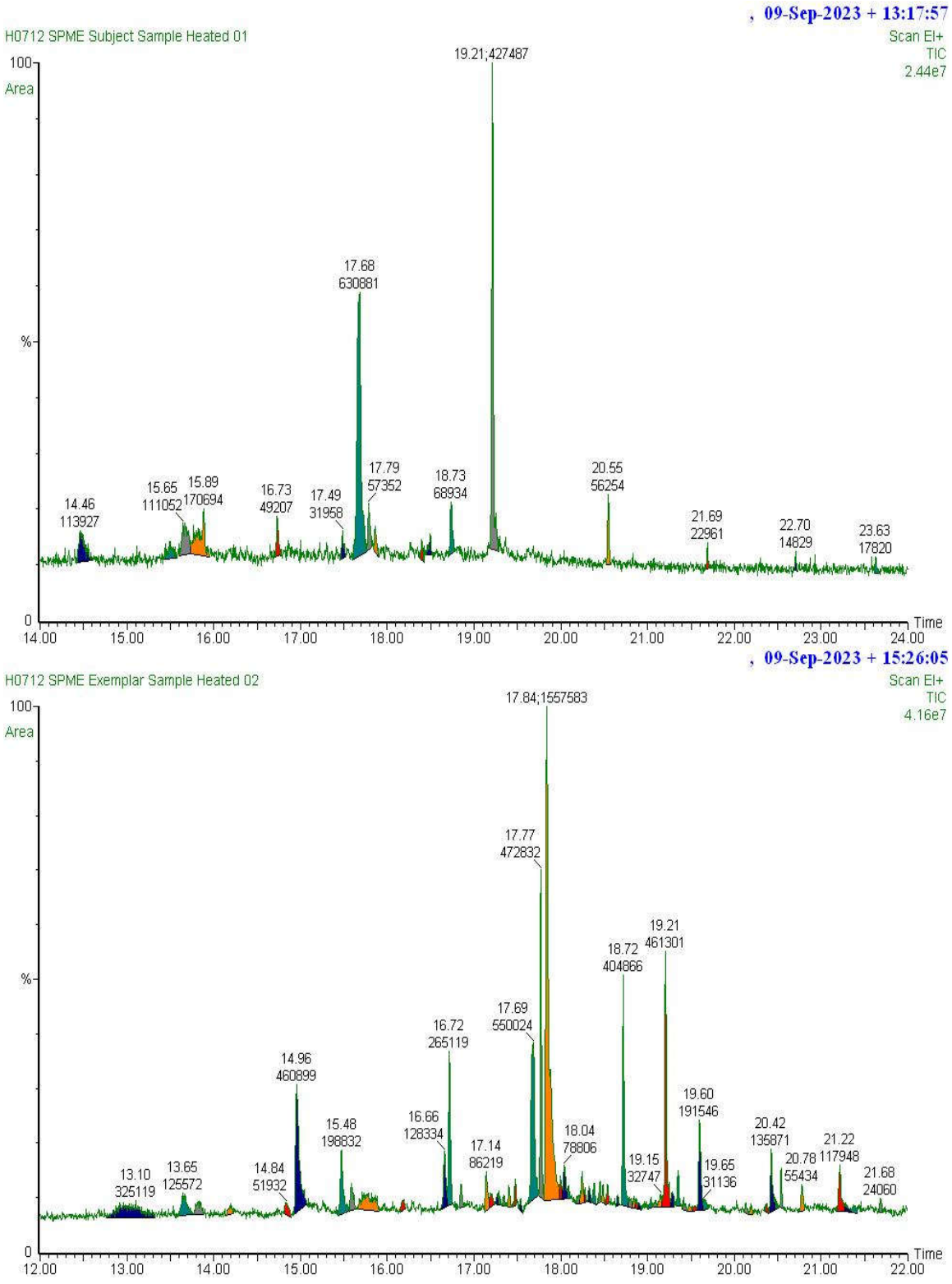

Figure 22:
Integrated total ion chromatograms of the heated subject (top) and exemplar (bottom) SPME analyses.

Materials Engineering, Inc.

Laboratory Accreditation

Materials Engineering, Inc., meets the requirements of ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories" (equivalent to relevant requirements of the ISO 9000 series of standards) as audited and accredited by an independent laboratory accreditation agency.

The accreditation covers specific tests and types of tests as represented on the scope of accreditation, which will be provided to customers upon request, or on the MEi website, www.materials-engr.com.

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2. The test data reported pertains only to the item(s) tested, and does not necessarily extend to the lot or batch from which the sample(s) were drawn. We accept no responsibility or liability for results due to non-representative test items, improper sampling, insufficient testing or misinformation.

3. Materials Engineering, Inc. based the findings, conclusions and opinions expressed in this report on the information provided by the customer and observations obtained at the time of inspection and preparation of this report. We reserve the right to add to, or modify this report should additional information become available.

4. Decision Rule - All statements of conformity shall be based on simple acceptance criteria – i.e., for the values produced by the analysis only, no uncertainty value shall be applied. If an ASTM specification is listed as a test method, the uncertainty of the measurement is included in the referenced test specification. For all other tests, contact the author of the report for an estimate of uncertainty.

5. It is our policy to retain components, samples and remnants for 30 days from the date of the report, after which they may be discarded. Please contact us immediately if you desire your components or samples to be returned to you, or to be retained for a longer period of time.

6. If you have any comments or concerns regarding the laboratory staff, the conduct of the testing and/or the report contents, we invite you to email the laboratory owners at customer.comment@materials-engr.com.