NATIONAL TRANSPORTATION SAFETY BOARD

Office of Research and Engineering
Materials Laboratory Division
Washington, D.C. 20594

June 24, 2021

MATERIALS LABORATORY FACTUAL REPORT Report No. 21-022

A. ACCIDENT INFORMATION

Place	:	Corpus Christi
Date	:	August 21, 2020
Vehicle	:	16-inch propane pipeline
NTSB No.	:	DCA20FM026
Investigator	:	Paul Stancil

B. COMPONENTS EXAMINED

The following pieces were submitted for examination:

(a) approximately 14-foot 4-inch segment of a 16-inch nominal diameter pipe with a concrete exterior coating
(b) two halves of a 1-foot in length concrete exterior coating that separated on-site from the west end of the pipe segment
(c) three teeth (designation “B3”, “C1”, and “E3”) that were disassembled from the cutterhead assembly and their corresponding keys[1]
(d) 50-pound, 17”x13”x3”, piece of debris found in cutterhead suction pipe (ladder cleanout)[2]
(e) 6-pound, 9”x8”x2”, piece of debris found in the cutterhead (main) pump strainer
(f) 2-pound, 4”x4”x2”, piece of debris found in the cutterhead (main) pump strainer
(g) 1-pound, 3”x2”x1”, piece of debris found in the cutterhead pump strainer
(h) 20-pound, 13”x12”x4”, piece of debris found on the port-side deck of the dredge

C. DETAILS OF THE EXAMINATION

1.0 On-site Work

The damaged pipe segment was submerged at the time of the accident and was recovered using the NTSB Pipe Recovery Protocol in Appendix 1. Commercial divers hired by Enterprise performed initial assessment of the damaged pipe. According to the divers, the damaged portion of the pipe contained two holes and two dents (as shown in Appendix 1). The pipe contained a concrete exterior coating. Concrete exterior coating had fractured and was missing from the damaged area of the pipe. A 14-foot 4-inch pipe segment of the pipe containing the damage area was cut and pulled out of the water. Approximately a 1-foot length portion of the concrete coating on the west end fractured into two halves during

[1] A key is a locking device that comprises of a tapered metal shaft and a rubber grommet that secures a tooth to a cutterhead blade.
[2] The symbol (“) indicates a unit of measure in inches.

the underwater recovery operation. The excised pipe segment and the two fractured halves of the concrete exterior coating from the west end of the excised pipe segment were transported to the NTSB Materials Laboratory for examination.

2.0 As-received

Figures 1 through 4 show photographs of the as-received pipe segment; two halves of 1-foot in length concrete exterior coating that separated from the west end of the pipe segment; three teeth that were removed from the cutterhead assembly; and five pieces of debris. The outer surface of the pipe near the girth weld was marked with a yellow marker by on-site divers with the 12 o'clock, 3 o'clock, 6 o'clock, and 9 o'clock positions. All clock positions were reported when looking east (flow direction of liquified propane) where 12 o'clock represented the top dead center (TDC). Both cut ends of the pipe at the inner surface were marked on-site with a white permanent marker that precisely indicated the 6 o'clock position (see Appendix 2). At the NTSB Materials Laboratory, a longitudinal line was drawn on the outer surface with a black marker from one end of the pipe to the other indicating the four clock positions, using the 6 o'clock mark on the inner surface as the reference for all the clock positions.

3.0 Safety Board Materials Laboratory Examination

Between December 7 and 10, 2020, a laboratory group examination of the pipe segment, cutterhead teeth and debris was conducted at the NTSB Materials Laboratory in Washington, D.C. Participation in the group examination was restricted to NTSB investigators that worked at the Washington, DC, headquarters office, in keeping with NTSB social distancing guidelines to prevent the spread of the Covid-19 virus. The following NTSB staff participated in the examination:

Frank Zakar	Materials Laboratory Group Chairman	NTSB
Edward Komarnicki	Member	NTSB
Paul Stancil	Member	NTSB
Luke Wisniewski	Member	NTSB

The following party members to the investigation were briefed on a live-video conference call at the end of each day regarding the progress of the laboratory work.

Jeff Morton	Member	Enterprise Products
Nhan Truong	Member	Enterprise Products
Graham Kenyon	Member	Orion Group
Alvaro Rodriguez	Member	PHMSA
Ron Perez	Member	RRC
LCDR Sara Conrad	Member	USCG
LT Lars Okmark	Member	USCG

Thereafter, follow-up laboratory work continued without a group, and weekly reports on the status of the laboratory work was issued to party members.

4.0 Pipe Segment

4.1 Background

The pipeline was operated by Enterprise Products Operating LLC (Enterprise) and was transporting liquified propane at the time of the accident. The propane was flowing east. Damage to the pipe was on the segment of line TX219 that was located underwater. Records provided by Enterprise indicated the pipe was specified as American Petroleum Institute (API) Specification for High-Test Line Pipe 5LX, Grade X46, 16-inch nominal outside diameter, with a 0.219-inch nominal wall thickness. The pipe was installed in 1968. The manufacturer of the pipe and date of manufacture are unknown. The seam weld[3] was electric resistance weld (ERW).[4] Corrosion control of the pipe was augmented by impressed current cathodic protection.

The pipe contained evidence of two exterior coatings (see figure 5). The innermost exterior coating was black consistent with coal-tar and the outermost exterior coating was hard and contained a mixture of cement and gravel consistent with concrete. Steel wire extended out of the cut ends of the concrete coating (steel wire was used as a reinforcement to hold the concrete coating together). When viewing the inner surface of the pipe, the pipe segment that extended between the girth weld and the east cut end contained evidence of a longitudinal protrusion (approximately 0.15 inch wide) typical of a longitudinal ERW seam at 1:30 o'clock looking east.

4.2 Examination

The pipe segment contained evidence of two punctures, arbitrarily labelled "P1" and "P2" in figures 6 through 9. The punctures were located at approximately the 7 o'clock position. Each puncture contained a lip portion that exhibited inward bending deformation. The bending deformation at the base of the lip was referred to as a fold. The fold for each puncture was located at the upper side of the puncture (see figure 8). The fold followed a nearly straight imaginary line across the length of the lip, indicated by a dashed line in figures 8 and 21, referred to as a fold line.

The two punctures were connected by a fracture "T1", as shown in figures 7 through 9. The pipe also contained five elongated dent features arbitrarily labelled "G1" through "G5", and three dents arbitrarily labelled "D1" through "D3", as shown in figures 7 and 9. Elongated dent "G1" contained one major gouge within the dent. Elongated dents "G2" through "G5" were nearly parallel to each other and diagonally oriented with respect to the axial length of the pipe. The upper end of the elongated dents did not intersect (connect with) the two punctures.

[3] Seam weld - longitudinal or spiral weld in the pipe which is made in the pipe mill.
[4] Enterprise assumes that its pipe segments constructed prior to 1970 have low-frequency ERW pipe seams.

4.2.1 Condition of the Concrete and Coal-Tar Coatings

As much as a 10-foot 5-inch length portion of the concrete exterior coating had separated (fractured) from the pipe in the general area of the two punctures "P1" and "2".[5] This area is labelled "portion void of concrete exterior coating" in figures 1 and 2. The concrete exterior coating to the east of this area remained attached to the pipe. The two halves of a 1-foot in length concrete exterior coating that separated on-site from the west end of the pipe segment is labelled "concrete separated on-site" in figures 1 and 2. The NTSB Materials Laboratory verified that the 1-foot length portion of concrete exterior coating separated from the west end of the pipe segment (see Appendix 3). The portions of concrete exterior coating that remained attached to the pipe contained evidence of longitudinal and circumferential cracks, several of which are indicated by a dashed line in figures 1 and 2. Within the 10-foot 5-inch length portion void of concrete external coating, coal-tar coating had fractured from isolated areas of the pipe, leaving metal surfaces of the pipe exposed. The exposed metal surfaces were covered with a white layer of calcareous deposit.[6] The non-metallic portions of the pipe (coal-tar and concrete exterior coatings) were not covered with a calcareous deposit.

The outer surface of the pipe in the area void of concrete exterior coating was also covered with a light layer of green algae, whereas the outer surface of the concrete exterior coating that remained on the pipe was covered with green algae and other biological fouling (biofouling); all of which were dry when received by the NTSB Materials Laboratory.[7] The outer surface of the concrete exterior coating was covered with mussel-like shells (open and empty of content) in an isolated area that extended approximately between the 10 o'clock and 12 o'clock position and extended between 6.3 feet and 9 feet from the grith weld.

During laboratory handling, pieces of the calcareous deposits fractured from the outer surface of the pipe, exposing cluster of gouges on the metal surface of the pipe, as shown in figure 10. Figure 11 shows the outer surface of the pipe after a large amount of calcareous deposit was removed from a cluster of gouges associated with elongated dent "G5" and "G4". The thickness of the calcareous deposit layer measured between 0.026 inch and 0.04 inch.

4.2.2 Size and Location of the Mechanical Damage

In the as-received condition, prior to removing any external coating and calcareous deposits, the location and size of each mechanical damage[8] area was measured relative to axial (longitudinal) distance from the girth weld and the 6 o'clock position. The location and size of each mechanical damage is shown in tables 1 through table 4. In summary, the

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[5] Conditions prior to cutting and lifting the submerged pipe.
[6] Calcareous deposit is a layer of material typically consisting of a mixture of calcium carbonate and magnesium hydroxide.
[7] Biofouling - is the accumulation of microorganisms, plants, algae, or small animals where it is not wanted on surfaces that are submerged in water such as inlets, rivers, and ocean, causing degradation to the primary purpose of that item.
[8] Mechanical damage - a generic term used to describe combinations of dents, gouges, and/or cold work caused by the application of external forces. Mechanical damage can also include coating damage, movement of metal, and high residual stresses.

longitudinal length of punctures “P1” and “P2” measured approximately 4 inches and 6 inches, respectively. The two punctures were connected by a fracture “T1”. The length of the fracture measured approximately 4 inches. The size of the dents was based on the extent of deformation noted on the outer surface. Depth measurements were made by extending a straight edge along the length of the pipe, then measuring with a caliper the distance between the straight edge and the outer surface of the dent. The depth of dents “D1”, “”D2” and “D3” measured approximately 0.69 inch, 1.87 inches and 0.2 inch, respectively. The elongated dents were diagonally oriented. The length of each elongated dent was measured from one end of the dent to the other end of the dent. The length of elongated dents “G2” through “G5” measured approximately 5 inches, 5.5 inches, 3.25 inches, and 10 inches, respectively. The width of the elongated dents “G1” through “G5” were not clearly defined and were not measured. Elongated dent “G1” contained only one major gouge, so in table 3 both the length and width of the single gouge were reported.

4.2.3 Cutting Out the Damaged Area

A rectangle plate portion of the pipe measuring approximately 43 inches in length and 21 inches in circumference that contained the mechanical damage area was marked for cutting.[9] The proposed cut lines for the rectangle piece are shown by dashed lines in figure 12. The cut lines were made at least 5 inches away from a damage area. Fragments of the coal-tar coating were firmly adhering to the outer surface of the pipe at the proposed cutting locations. A plastic wedge or putty knife was forced between the pipe and coal-tar coating and calcareous deposits to assist removal. Difficult-to-remove coal-tar coating at isolated areas was removed by striking the coating with a mallet.

A drill hole was made through the wall at the upper west end corner of the proposed rectangle cut locations. This hole allowed a hand-held (portable) reciprocating saw to be inserted into the wall of the pipe to start a cut. A saw cut was made in the wall that extended approximately 9-inch longitudinally and 12-inch circumferentially from the drilled hole, respectively. The remaining portion of the rectangle plate was cut by plasma-torch. Figure 13 shows the inner surface of the excised rectangle plate portion prior to cleaning.

4.2.4 Exposing the Gouges and Dents

After the rectangle piece was cut from the pipe, the outer and inner surfaces were cleaned with Alconox, a commercial detergent, using a soft bristle brush. Figures 14 through 24 show photographs of the mechanical damage features when viewed from the outer and inner surface of the pipe after cleaning.

The cleaning procedure exposed all the gouges that were associated with elongated dents “G1” through “G5”. To reiterate, only one major gouge was found within dent “G1”, whereas a cluster of gouges was found with each respective elongated dent. Figure 11 shows an example of a cluster of gouges associated with elongated dent “G5”. The width of the cluster of gouges associated with elongated dent “G5” is indicated by a bracket in figure 11.

[9] Approximately between 19 inches and 63 inches from the girth weld and between 5:30 and 10:30 looking east.

Table 5 shows the measured length and width of the exposed cluster of gouges associated with each elongated dent. Table 6 shows the shortest distance between the upper-most end of the cluster of gouges associated with elongated dents “G1” through “G5” and the fold line of a puncture (“P1” and “P2”). The least distance between any cluster of gouges associated with elongated dents “G1” through “G5” and the fold line of a puncture measured 0.3 inches, and it was associated with cluster of gouges extending from elongated dent “G5”. The cluster of gouges associated with elongated dents “G1” through “G5” did not extend into the lip portion of a puncture. Also, the upper end of each elongated dent, “G1” through “G5”, did not connect or extend into a puncture.

The cleaning procedure revealed dent “D1” contained two major gouges (see figure 16), dent “D2” contained evidence of a cluster of gouges within the dent (see figure 17), and dent “D3” contained several minor gouge marks (see figure 18).

Examination of the lip portion for puncture “P1” revealed the outer surface exhibited evidence of impact marks (deformed surface that contained gouges). The impact marks were prevalent in the areas along the edges of the fracture faces, as shown in figure 23.

Examination of the lip portion for puncture “P2” also revealed the outer surface exhibited evidence of impact marks (deformed surface that contained gouges). The impact marks were prevalent in the areas along the edges of the fracture faces, as shown in figure 24. The east and west ends of the puncture exhibited evidence of severe metal deformation, in areas indicated by brackets in figures 20, 22 and 24. When looking at the inner face of the pipe, the east and west ends of the puncture “P2” showed inward bending deformation, in the general direction indicated by arrows in figure 24. A portion of the wall had fractured and was missing from puncture P2” in the area enclosed by a dashed line in figures 20, 22, 24, and 26 (the lip portion did not extend into the missing portion of the wall).

The fracture faces of the punctures “P1” and “P2” and fracture “T1” exhibited no evidence of a pre-existing crack, such as a fatigue crack, or a corrosion cavity along the fracture. The fracture faces showed evidence of a course texture features typical of overstress separation.

4.2.5 Chemical Composition of the Pipe

The 1968 edition of API Standard 5LX was consulted because it was the edition that was in effect at the time the pipe was installed. According to API Standard 5LX, the maximum amount of carbon, manganese, phosphorus, and sulfur, by percent, that is permitted in the steel is governed by the grade of steel, how the steel was processed (i.e., seamless versus welded, electric or open-hearth furnace). Chemical analysis was performed on a 5-inch longitudinal by 7-inch circumferential sample that was cut from the upper corner of the rectangle plate described in section 4.2.3. The chemical analysis was performed by Lehigh Testing laboratory, New Castle, Delaware. Table 7 shows the results of the chemical analysis for elemental content that was greater than 0.01% by weight. The results indicate that the chemical composition of the pipe met the criteria for specification API 5LX, Grade

X46, seamless and welded pipe, made by electric-furnace, open-hearth, basic-oxygen or killed deoxidized basic-bessemer process, and ordered as expanded or cold-expanded pipe.

4.2.6 Ultimate Tensile Strength of the Pipe

The 1968 issue of API Standard 5LX indicates that the ultimate tensile strength and yield strength for grade X46 pipe should be no less than 63,000 pounds per square inch (psi) and 46,000 psi, respectively. A circumferential-radial section was made through the wall of the pipe in the orientation indicated by section line “R-R” in figure 12. The section was placed in a metallurgical mount and the section was polished. Rockwell “B” hardness testing of the section produced a hardness between 86 HRB and 90 HRB. A total of five readings were made and the average hardness was calculated to be 89 HRB. The average hardness value was converted to an approximate ultimate tensile strength (UTS) value by using conversion tables in ASTM A-370, *Standard Method and Definition for Mechanical Testing of Steel Products*. The average hardness value converted to a UTS of approximately 88,000 psi. The minimum UTS specified for the pipe is 63,000 psi.

4.2.7 Dimensions of the Pipe

The thickness of the pipe wall was measured on metallurgical section “R-R” in figure 12 prior to encasing the section in a mount. The wall thickness measured between approximately 0.22 inch, which was within the specified tolerances for 0.219-inch wall thickness pipe (0.192 and 0.252 inch). At the east end of the pipe, the thickness of the coal tar coating measured approximately 0.2 inches and the thickness of the concrete coating measured approximately 2.4 inches.

5.0 Fit Test (Inserting a Tooth into a Puncture)

The tip portion of tooth “C1” was placed on the outer surface of the pipe in the area adjacent to the larger puncture “P2”. The tip portion was then partially inserted into the puncture (see figure 25). When the tooth was inserted into the puncture, there was room for the entire width portion at the tip to enter the puncture (see figure 26).

The fitting procedure was repeated for the smaller puncture “P1”. The tip portion of tooth “C1” was placed on the outer surface and adjacent to puncture “P1” (see figure 27). Only a tip corner portion of the tooth and a portion of the tooth width (approximately 50% of the width portion of the tooth) extended into the puncture (see figure 28).

The tip portion of tooth “C1” was placed on the outer surface of the pipe in the area adjacent to dent “D2” (see figure 29). The width of the tooth was specified as 109 millimeters (4.3 inches). The width of the cluster of gouges associated with dent “D2” measured as wide as approximately 4.4 inches.

6.0 Teeth[10]

The teeth and cutterhead assembly were manufactured by Vosta LMG in the Netherlands. The teeth specified for the cutterhead assembly was part number SC15.01, referred to in the Vosta Maintenance Manual as “wide chisel” teeth. The same manual indicated that the width of the tooth was specified as 109 millimeters (4.3 inches) and that each tooth weighs about 8.5 kilogram (18.7 pounds). Welding repair of the tooth is prohibited according to the same maintenance manual. The material for the tooth was specified as high alloy cast steel. A representative from Vosta indicated that the specified alloy composition of the teeth was company proprietary information (due to competitive market among companies supplying products that are wear and abrasion-resistant).

The submitted teeth were ultrasonic cleaned in Alconox, a commercial detergent. For the purpose of this report, the face showing the part number and name of the manufacturer is referred to as the front face and the opposite face is referred to as the back face. Figure 30 shows a photograph of the front face of teeth “C1” and “E3”. Figure 31 shows oblique views of the front face of tooth “C1” from the left and right edges. Visual examination of the teeth revealed the front face of intact teeth “C1” and “E3” were marked with the part number “SC15.01” and the name of the manufacturer “VOSTA”. The tip portion of each tooth contained evidence of wear (abrasion) marks. A deformed metal fragment was embedded at the tip and back side of tooth “C1” (see figures 32 through 34).

Chemical analysis was performed on a sample of tooth “C1” and “E3” by Lehigh Testing laboratory, New Castle, Delaware. Table 8 shows the results of the chemical analysis for elemental content that was greater than 0.01% by weight.

7.0 Metal Transfer

7.1 Checking for Metal Transfer on the Outer Surface of the Pipe

A metallurgical section was made through a cluster of gouges on the outer surface of the pipe that was associated with elongated dent “G5”, in the area indicated by section line “Y-Y” in figure 15. Prior to encasing the section in a metallurgical mount, X-ray energy dispersive spectroscopy (EDS) analysis of the cut surface of the pipe wall produced a spectrum that contained a major elemental peak of iron and minor elemental peaks of manganese and carbon, consistent with plain carbon steel (see figure 35). This spectrum established a baseline for the material of the pipe. Also, prior to encasing the section in a metallurgical mount, EDS analysis of the gouges on the outer face of the pipe produced a spectrum that contained the same elemental peaks as in the pipe and additional elemental peaks of chromium, silicon, magnesium, and oxygen (see figure 36). The two over-lapping spectra are shown in figure 37. Magnesium is an element that is found in calcareous deposits. Oxygen is present as an oxide.

[10] See NTSB Materials Laboratory Factual Report No. 21-023 for additional details regarding the on-site inspection of the cutterhead assembly and teeth.

Section “Y-Y” was placed in a metallurgical mount and polished. The section was etched with 2% Nital reagent. The wall of the pipe contained a microstructure of pearlite and ferrite grains, typical for a carbon steel (see figure 38). The outer surface of the wall showed evidence of deformed grains and metal flow (see figure 39). EDS spectrum of the deformed grains at the outer surface in several isolated areas contained the same elemental peaks as in the pipe and additional elemental peaks of chromium and silicon.

7.2 Checking for Metal Transfer at the Tip of Tooth “C1”

A saw cut was made through the tip of tooth “C1” across the entire width of the tooth that incorporated the deformed metal fragment (see cut lines in figure 32). EDS analysis of the cut face of the tooth produced a spectrum that contained a major elemental peak of iron and minor elemental peaks of chromium, silicon, manganese, and carbon (see figure 40). This spectrum was used as a baseline that represented material of the tooth. The EDS analysis of the deformed metal fragment (see figure 41) produced a spectrum that contained a major elemental peak of iron and minor elemental peaks of silicon, manganese, and carbon. The EDS spectrum of the deformed metal fragment showed no evidence of a chromium and silicon elemental peaks. Figure 42 shows the overlapping EDS spectra.

A metallurgical section was made through the tip of the tooth and deformed metal fragment, in the orientation indicated by section line “X-X” in figure 33. Figure 43 shows an overall view of section “X-X”. The section was prepared and etched with 2% Nital reagent. Examination of the prepared section revealed the deformed metal fragment showed evidence of severely deformed microstructure of ferrite and pearlite grains (see figure 44), whereas the tooth showed evidence of a cast microstructure (see figure 45).

The tip portion of tooth “C1” also contained evidence of metal transfer in an isolated area indicated by arrow “A” in figure 34. EDS spectrum of metal build-up in the area indicated by arrow “A” was similar to the EDS spectrum of the pipe.

8.0 Teeth “B3” and “E3”

The tip portion of tooth “B3” was fractured and only the base portion was submitted. The tip portion of tooth “B3” was missing and was not located during underwater recovery of the cutterhead assembly or removal of the pipe segment. The fracture face of tooth “B3” showed coarse fracture features typical of overstress separation with no evidence of a preexisting fracture, such as a fatigue crack.

Bench binocular microscope examination of tooth “E3” revealed wear at the tip portion. Based solely on visual examination, there was no clear indication whether the wear surface contained metal that was foreign to the tooth. EDS analysis was not performed at the tip portion of tooth “E3”.

9.0 Analysis of the Calcareous Deposit

The outer surface of a calcareous deposit was covered with green algae, as shown in Appendix 4. X-ray fluorescence (XRF) analysis was performed on the sample of the calcareous deposit to determine the elements that were present and their relative amounts (reported in weight percent). The analysis was performed by Eurofins EAG Materials Science, LLC, Sunnyvale, California. The results of the analysis showed that the calcareous deposit sample consisted primarily of 18.7% magnesium, 12.8% calcium, 54.8% oxygen, and 12.8% carbon with trace levels of a number of other elements. Appendix 5 shows the details of the XRF analysis.

X-ray diffraction (XRD) analysis was performed on a sample of the calcareous deposit to determine the crystalline phases that were present and their relative amounts (reported in weight percent). Compound such as calcium carbonate (chemical formula $CaCO_3$) can exist in different crystalline structures. For example, calcium carbonate can exist as an aragonite crystalline structure or as a calcite crystalline structure. XRD analysis can identify these different crystalline phases. The results of the XRD analysis showed that major constituents in the calcareous deposit were primarily 52.9% Brucite [$Mg(OH)_2$]; 38.2% Aragonite [$CaCO_3$]; 4.0% Calcite [$CaCO_3$]; and 3.9% Quartz [SiO_2]. The chemical formula for each identified phase is enclosed by brackets. Appendix 6 shows the details of the XRD analysis.

10.0 Fragments of the Concrete Exterior Coating

The 1-pound, 2-pound, and 6-pound debris appeared white and exhibited a smooth texture that was similar to the concrete exterior coating on the pipe. These three debris exhibited a slightly round curvature. The thickness of each piece was uniform along their length, each measuring approximately 2 inches, consistent with the thickness of the concrete exterior coating (approximately 2 inches).

The 20-pound debris appeared gray. The outer surface contained a circumferential ribbed pattern and the inner surface exhibited a smooth and round surface. The thickness of the debris varied between 2.1 inches at the valley and 2.2 inches at the peak portion of each rib pattern. The thickness of this pieces was not 4 inches as reported in the submission paperwork.

The 50-pound debris exhibited a round curvature. The debris was covered with barnacles and other hard shells. The thickness of the debris measured approximately 3 inches.

An attempt was made to match the fracture faces of each debris to the fracture face of the concrete exterior coating that was adhering to the pipe. The fracture faces of the submitted concrete debris pieces did match each other or the concrete exterior coating on the pipe segment.

Prepared by:

Frank Zakar
Senior Metallurgist

TABLE 1
Measured Size and Location of Punctures, Dents, and a Fracture,
Before Removing Calcareous Deposit

Feature	Depth (inch)	Horizontal (Axial) Distance to the Girth Weld (inch)			Total Axial Length (inch)	Vertical (Circumferential) Distance from 6:00 o'clock Position (inch)			Measured Length of a Fold Line (inch)	Clock Position Looking East (o'clock)
		West End	Center	East End		Bottom End	Center	Top End		
Puncture (P1)	N/A	31.3	33.3	35.3	4.0	3.5	4.3	5.0	3.0	7:00
Puncture (P2)	N/A	39.3	41.7	45.5	6.2	2.5	4.0	6.0	5.0	7:00
Dent (D1)	0.7	36.0	37.5	39.0	3.0	1.1	1.5	2.0	N/A	6:15
Dent (D2)	1.9	47.5	49.8	53.5	6.0	4.3	6.3	9.0	N/A	8:00
Dent (D3)	0.2	56.5	57.0	57.8	1.3	8.0	8.5	9.0	N/A	8:00
Fracture (T1)	N/A	35.3	37.0	39.3	4.0	4	4	4	N/A	7:00

Note: N/A – not applicable

TABLE 2
LOCATION OF ELONGATED DENTS,
BEFORE REMOVING CALCAREOUS DEPOSITS
(INCH)

FEATURE	HORIZONTAL (AXIAL) DISTANCE TO THE GIRTH WELD			VERTICAL (CIRCUMFERENTAIL) DISTANCE FROM 6:00 O'CLOCK		
	WEST END	CENTER	EAST END	BOTTOM END	CENTER	TOP END
ELONGATED DENT (G1)	26.5	27.0	27.5	2.5	2.6	2.8
ELONGATED DENT (G2)	25.5	27.0	29.5	5.0	7.0	8.3
ELONGATED DENT (G3)	29.0	30.0	32.5	6.3	9.0	11.0
ELONGATED DENT (G4)	33.8	34.8	36.0	9.5	10.5	12.0
ELONGATED DENT (G5)	36.5	40	43.0	7.0	10.0	13.5

TABLE 3 SIZE OF ELONGATED DENTS BEFORE REMOVING CALCAREOUS DEPOSITS				
FEATURE	DEPTH	MEASURED LENGTH (END-TO-END)	MEASURED WIDTH	CLOCK POSITION LOOKING EAST, RANGE
	(INCH)	(INCH)	(INCH)	(O'CLOCK)
ELONGATED DENT (G1)	0.1	1.3	0.5	6:30
ELONGATED DENT (G2)	0.6	5.0	Not clear*	7:15 – 8:00
ELONGATED DENT (G3)	0.7	5.5	Not clear*	7:15 – 8:30
ELONGATED DENT (G4)	0.3	3.3	Not clear*	7:30 – 9:00
ELONGATED DENT (G5)	0.8	10.0	Not clear*	7:30 – 9:15

(*) – No measurement was made because the outline/border was not clearly defined.

TABLE 4. MEASURED DISTANCE BETWEEN ELONGATED DENTS, <u>BEFORE</u> REMOVING CALCAREOUS DEPOSITS (INCH)		
ELONGATED DENTS OF INTEREST	CENTER-TO-CENTER	BORDER OF ONE DENT TO THE ADJACENT BORDER OF THE OTHER DENT
BETWEEN G1 AND G2	3.5	Not clear*
BETWEEN G2 AND G3	3.0	Not clear*
BETWEEN G3 AND G4	5.0	Not clear*
BETWEEN G4 AND G5	3.0	Not clear*

(*) – No measurement was made because the outline/border was not clearly defined.

TABLE 5 SIZE OF EXPOSED CLUSTER OF GOUGES <u>AFTER</u> REMOVING CALCAREOUS DEPOSITS			
FEATURE	MEASURED LENGTH (END-TO-END)	MEASURED WIDTH	CLOCK POSITION, RANGE
	(INCH)	(INCH)	LOOKING EAST
ELONGATED DENT (G1)*	1.3	0.3	Similar to Table 3
ELONGATED DENT (G2)	6.1	0.7	Similar to Table 3
ELONGATED DENT (G3)	8.0	4.0	Similar to Table 3
ELONGATED DENT (G4)	7.5	1.6	Similar to Table 3
ELONGATED DENT (G5)	11.0	3.2	Similar to Table 3

Note(*) – Elongated dent "G1" contained only one major gouge and only the length and width of the major gouge is reported.

TABLE 6. Measured Shortest Distance between Upper End of Gouges and a Puncture (inches)	
Distance between Upper End of G1 and P1	3.7
Distance between Upper End of G2 and P1	1.5
Distance between Upper End of G3 and P1	3.5
Distance between Upper End of G4 and P2	3.5
Distance between Upper End of G5 and P2	0.3

Table 7.
Chemical Composition for API 5LX, Grade X46 Pipe, 1968 edition
Specified Maximum Limits
(Weight %)

Element	Seamless				Welded				Measured
	Electric-furnace, open-hearth, basic-oxygen or killed deoxidized basic-bessemer		Killed Deoxidized acid-bessemer or killed deoxidized basic-bessemer		Electric-furnace, open-hearth, basic-oxygen or killed deoxidized basic-bessemer		Killed Deoxidized acid-bessemer or killed deoxidized basic-bessemer		
	Non-expanded	Cold-expanded	Non-expanded	Cold-expanded	Non-expanded	Cold-expanded	Non-expanded	Cold-expanded	
Aluminum	-	-	-	-	-	-	-	-	<0.01
Arsenic	-	-	-	-	-	-	-	-	0.009
Carbon	0.31	0.29	0.27	0.24	0.30	0.28	0.26	0.23	0.28
Cobalt	-	-	-	-	-	-	-	-	<0.01
Chromium	-	-	-	-	-	-	-	-	0.03
Copper	-	-	-	-	-	-	-	-	0.26
Manganese	1.35	1.25	1.35	1.25	1.35	1.25	1.35	1.25	1.23
Molybdenum	-	-	-	-	-	-	-	-	0.02
Niobium [11]	-	-	-	-	-	-	-	-	<0.01
Nickel	-	-	-	-	-	-	-	-	0.04
Phosphorus	0.04	0.04	0.10	0.10	0.04	0.04	0.10	0.10	0.029
Sulfur	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.016
Silicon	-	-	-	-	-	-	-	-	0.01
Titanium	-	-	-	-	-	-	-	-	<0.01
Vanadium	-	-	-	-	-	-	-	-	<0.01
Iron	Remainder	Remainder	Remainder	Remainder	Remainder	Remainder	Remainder	Remainder	Remainder

Note: (“-“) indicates not specified

[11] Formerly known as the element columbium.

Table 8. Chemical Composition of Teeth (Weight %)		
Element	Tooth “C1” (Measured)	Tooth “E3” (Measured)
Aluminum	0.03	0.03
Arsenic	0.011	<0.008
Carbon	0.23	0.24
Cobalt	0.01	0.01
Chromium	1.39	1.39
Copper	0.10	0.10
Manganese	1.16	1.12
Molybdenum	0.20	0.20
Niobium	0.01	0.01
Nickel	0.46	0.46
Phosphorus	0.012	0.012
Sulfur	0.009	0.009
Silicon	1.56	1.52
Titanium	0.07	0.06
Vanadium	0.02	0.02
Iron	Remainder	Remainder

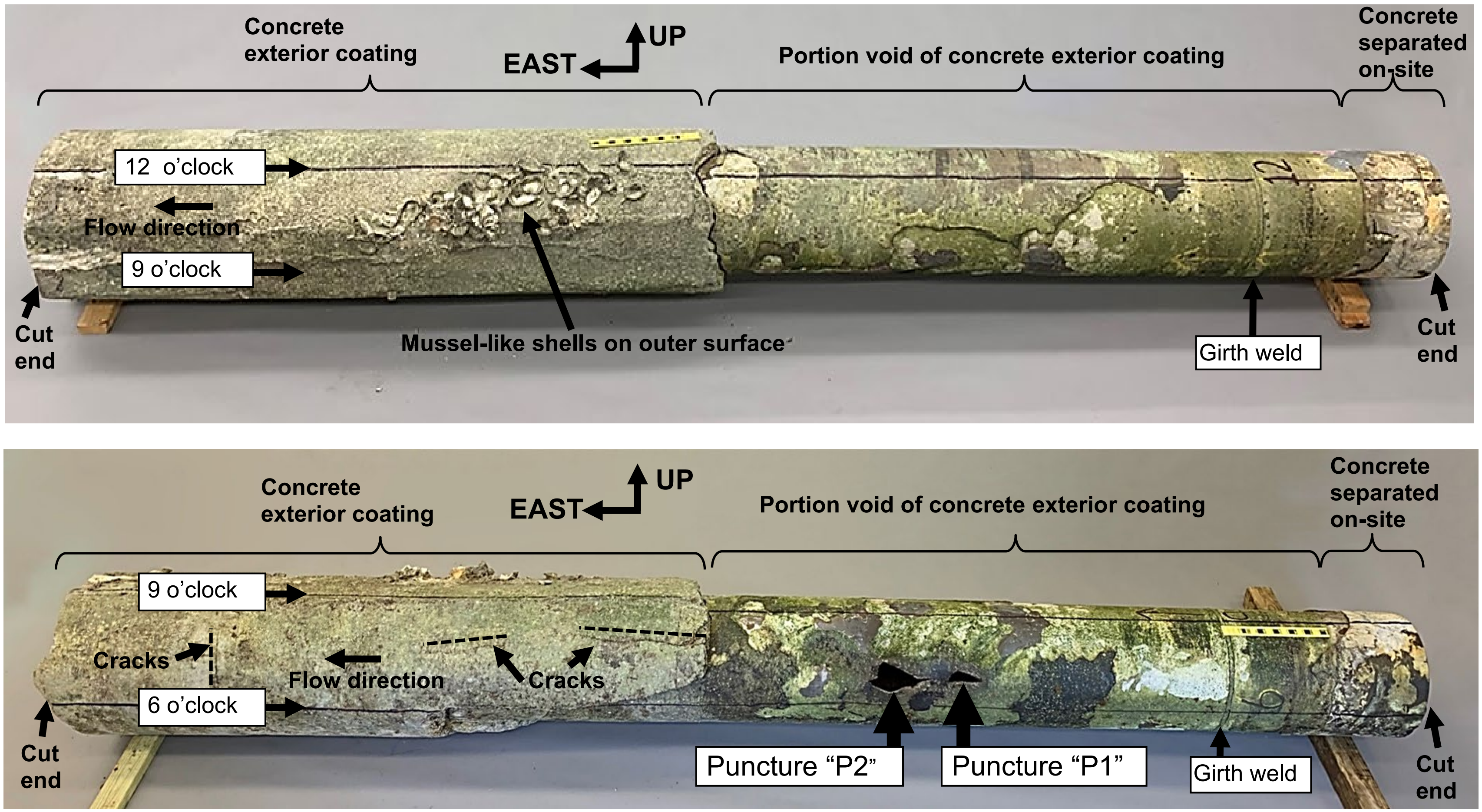


Figure 1. As-received damaged pipe segment showing the top side (12 o'clock) and the bottom side (6 o'clock). The pipe contained two punctures in the areas indicated by arrows "P1" and "P2". The punctures were located at approximately at 7 o'clock. Clock positions are when looking east. Cracks on the concrete external coating are indicated by dashed lines. Two halves of a 1-foot in length concrete exterior coating separated on-site (during recovery operation) from the west end of the pipe segment.

Figure 2. Two halves of 1-foot in length concrete exterior coating that separated on-site from the west end of the pipe segment.

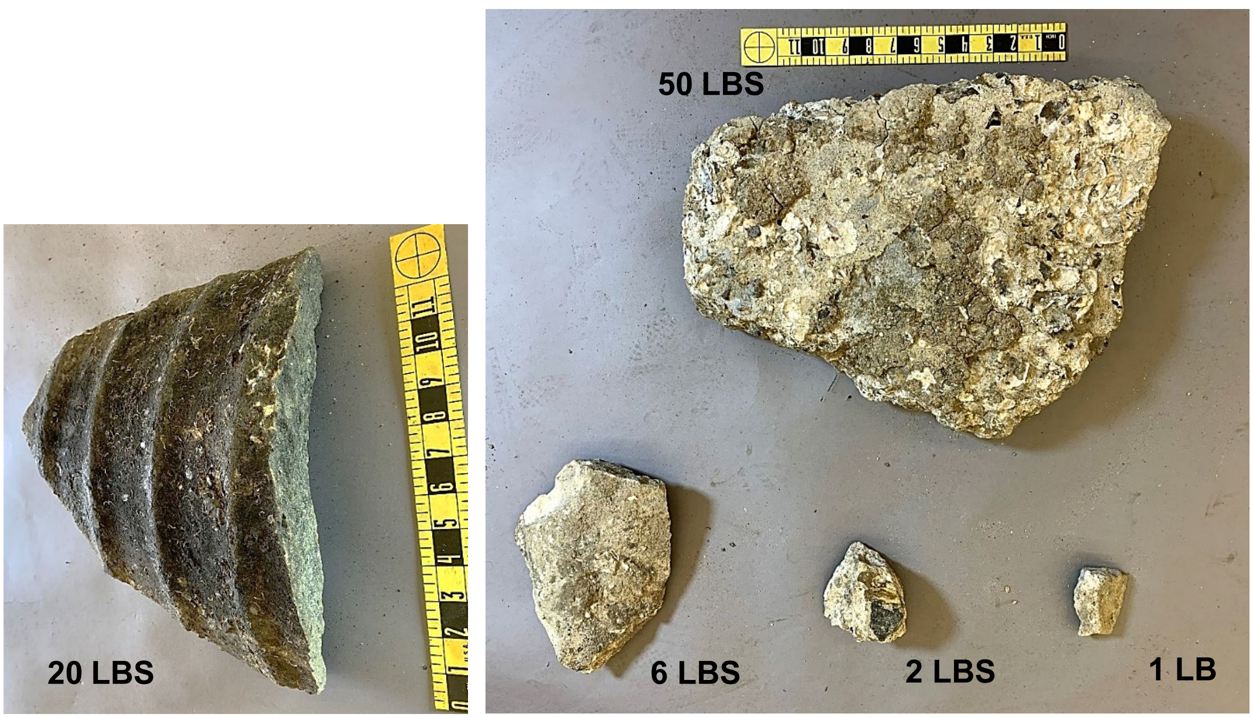


Figure 3. Five pieces of debris that were submitted and their respective weights in pounds (lbs).

Figure 4. As-received fractured tooth "B3", intact teeth "E3" and "C1", and corresponding keys that were disassembled from the cutterhead assembly, respectively.

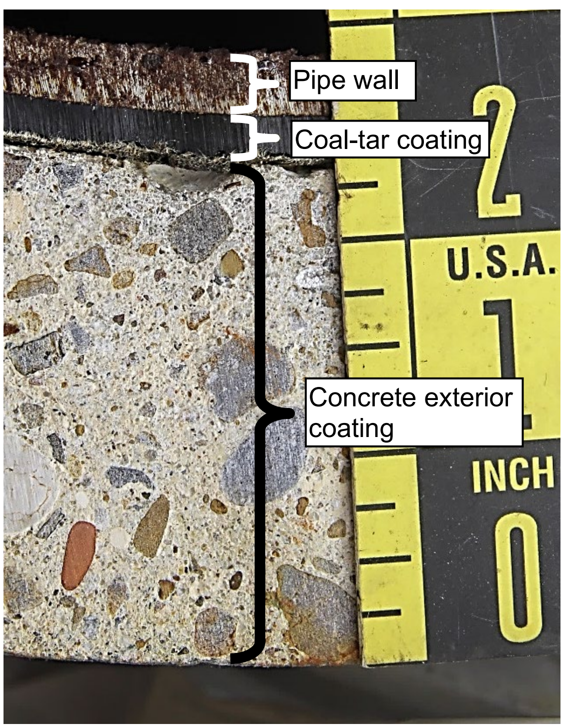


Figure 5. As-received fractured tooth "B3", intact teeth "E3" and "C1", and corresponding keys that were disassembled from the cutterhead assembly, respectively.

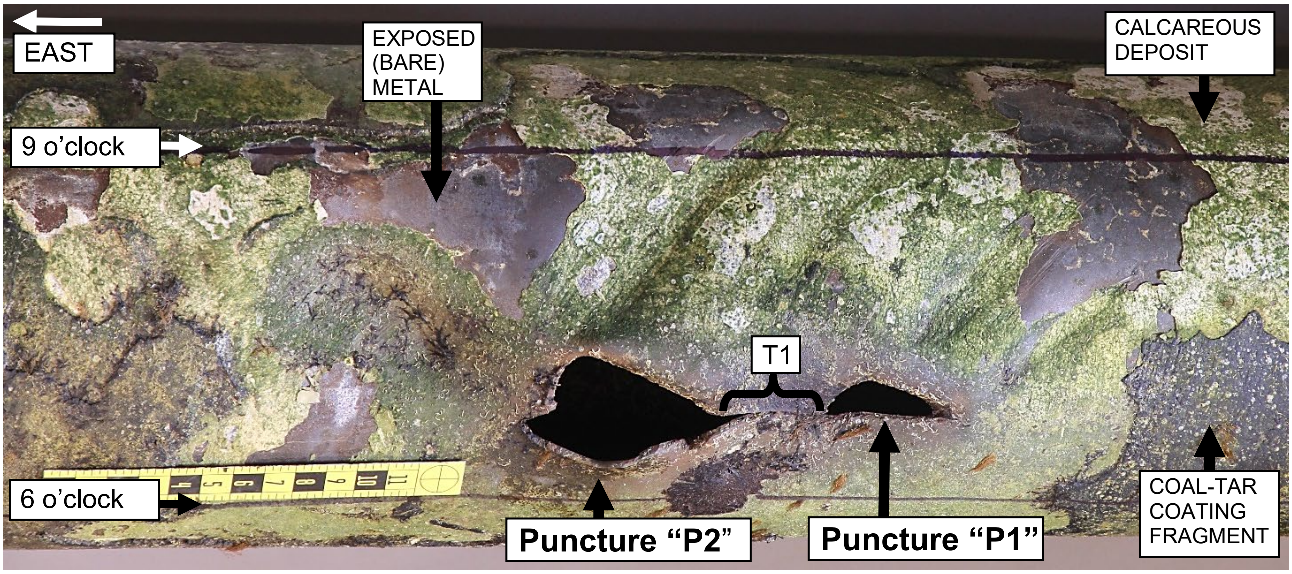


Figure 6. Close-up view of the as-received pipe in the areas of punctures "P1" and "P2" and a fracture, indicated by bracket "T1" that intersected both punctures. During handling, calcareous deposit separated from isolated areas of the pipe exposing (bare) metal.

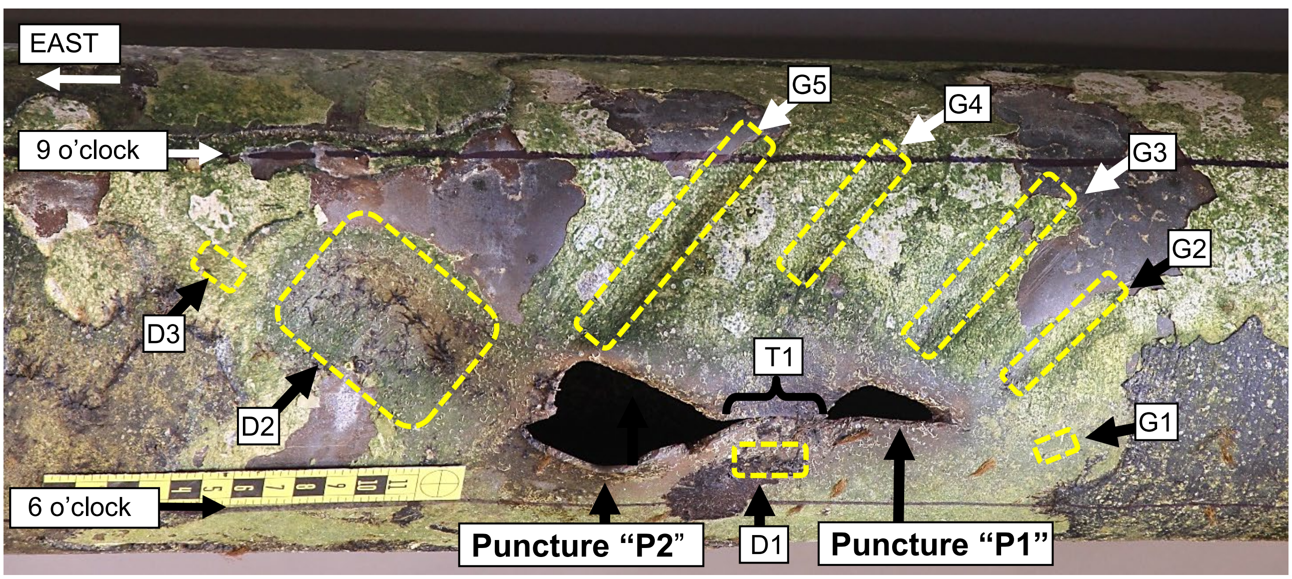


Figure 7. Same view as in figure 6 but also showing the location of elongated dents "G1" through "G5"; and dents "D1" through "D3", which have been highlighted by yellow dashed lines.

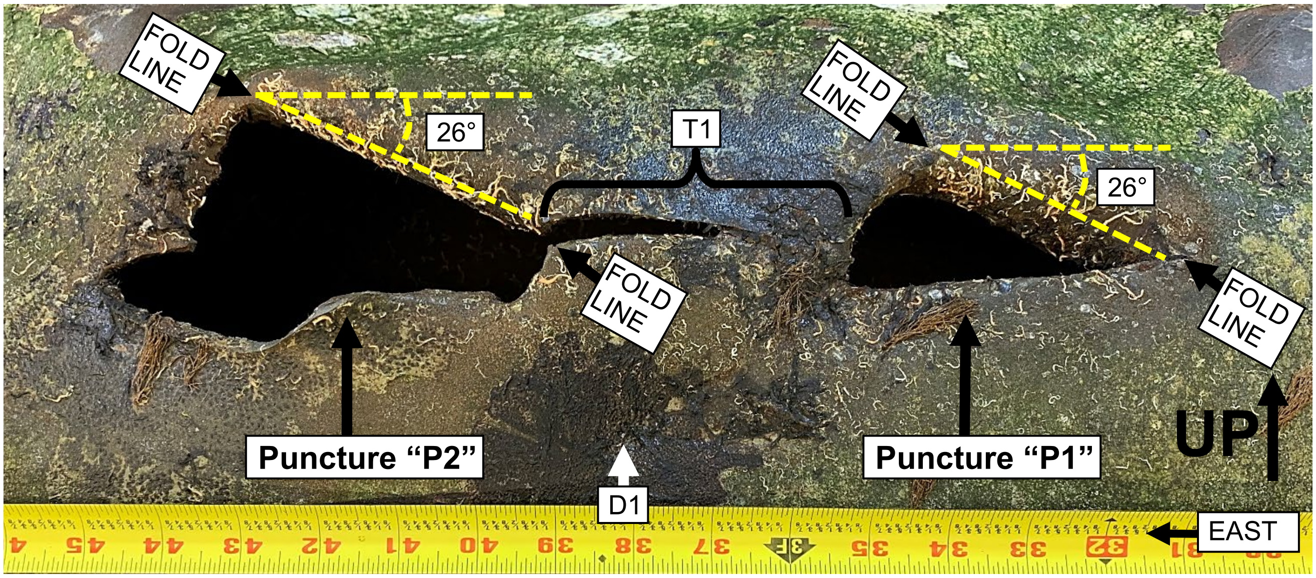


Figure 8. Close-up view of the pipe in the area of punctures "P1" and "P2", fracture "T1", and dent "D1". Each puncture contained a lip (wall) portion that exhibited inward bending deformation. Only a small portion of each lip is visible in this view. The base of the lip (containing a fold line) for each puncture was located at the upper end of the puncture. The outer surface was covered with biological fouling (biofouling). Oblique view of the imaginary fold line at the base portion of the lip for puncture "P1" is shown in figure 21. The fold lines were oriented approximately 26 degrees down relative to the axial length of the pipe.

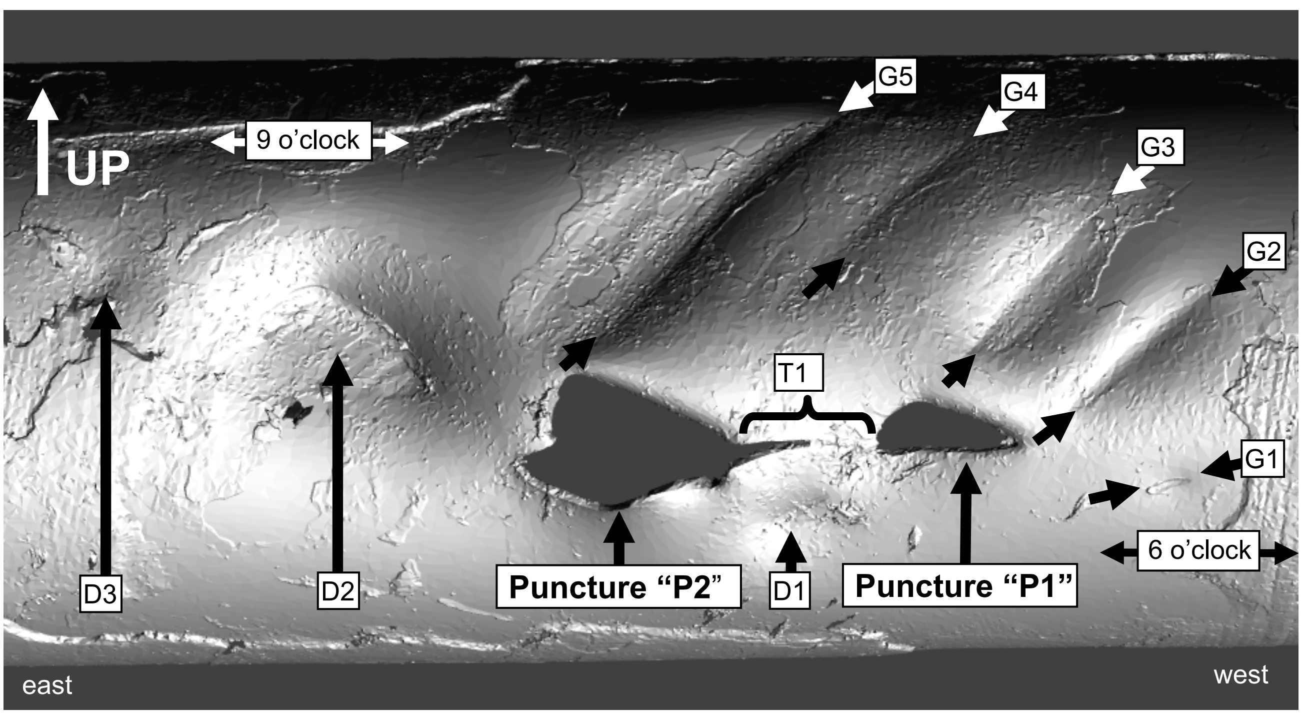


Figure 9. 3D laser scanned image of the pipe in the area of punctures "P1" and "P2", fracture "T1", elongated dents "G1" through "G5", and dents "D1" through "D3". Scanned onsite by contractor for Enterprise. Courtesy of Enterprise.

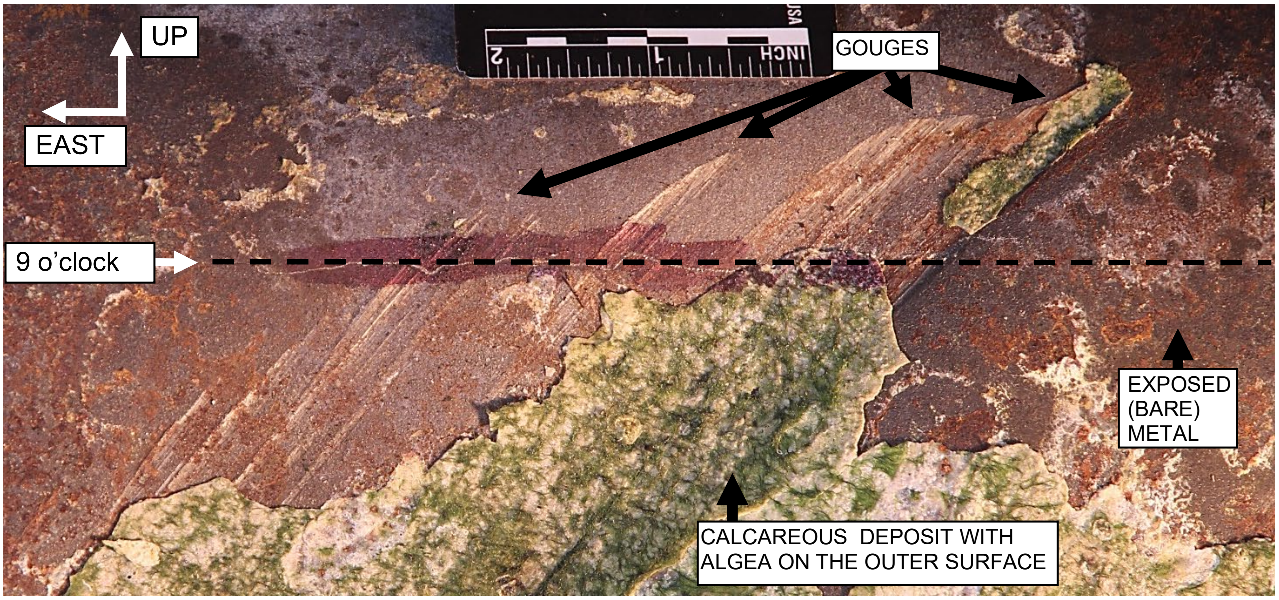


Figure 10. Close-up photograph of a cluster gouges on the metal surface of the pipe in the general area at the upper end and within elongated dent "G5". Cluster of gouges extended under the adhering and intact calcareous deposit.

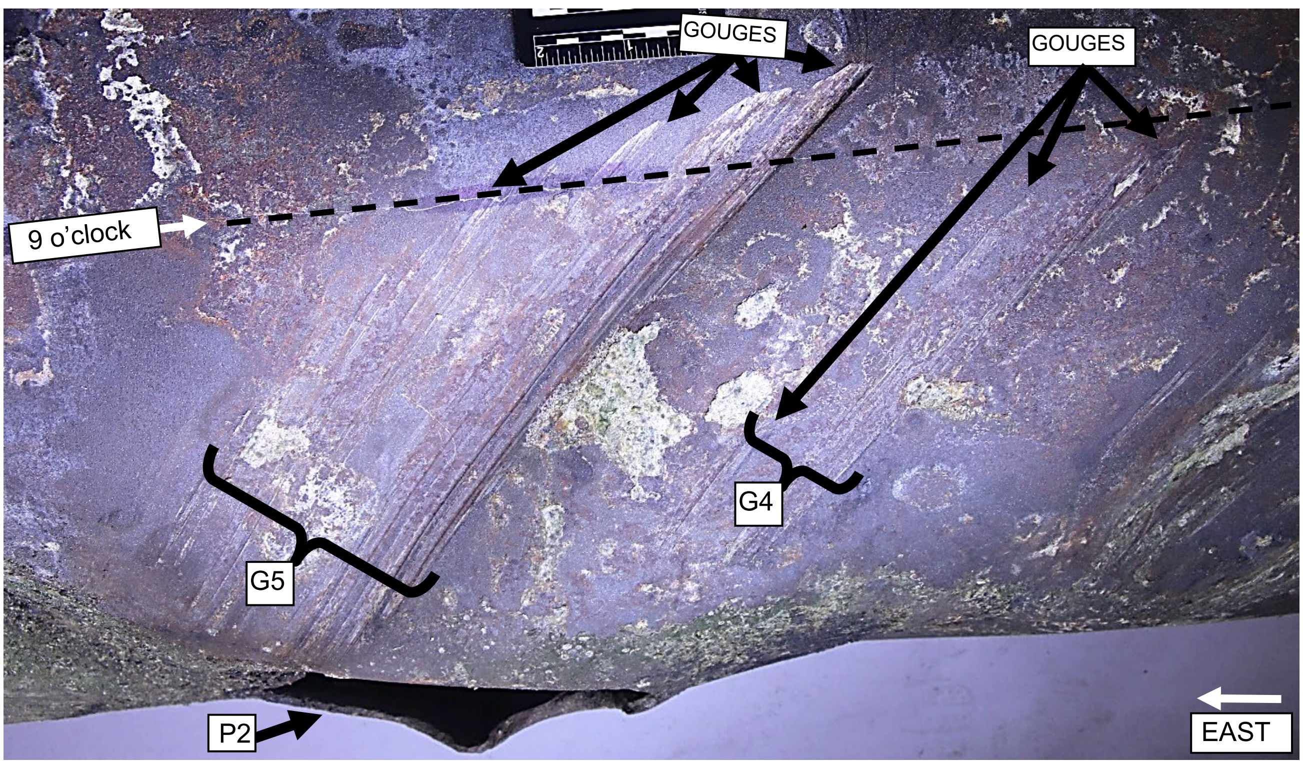


Figure 11. Exterior view of the pipe in the area of puncture "P2" after a large amount of calcareous deposit was removed from elongated dents "G4" and "G5" showing exposed cluster of gouges. The width portion of each cluster gouges is indicated by a bracket. A cluster of gouges was associated with each elongated dent. The width associated with each elongated dent was measured and recorded in table 5.

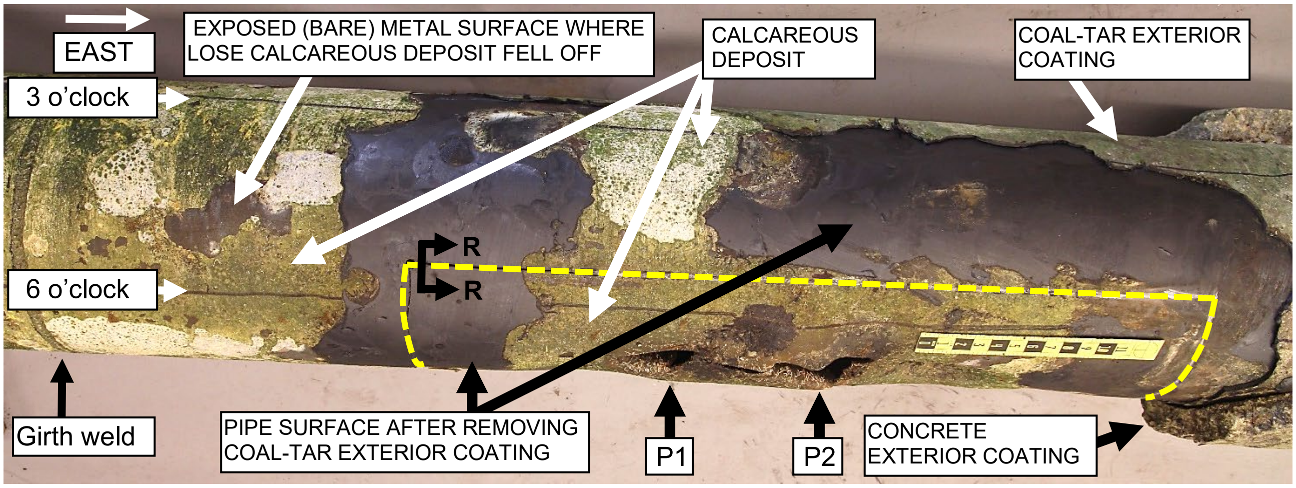


Figure 12. Exterior view of the pipe showing the damage area and proposed cut lines indicated by dashed lines. The black areas on the outer surface of the pipe represent areas where the coal-tar exterior coating was removed prior to cutting the rectangle piece containing the damage area.

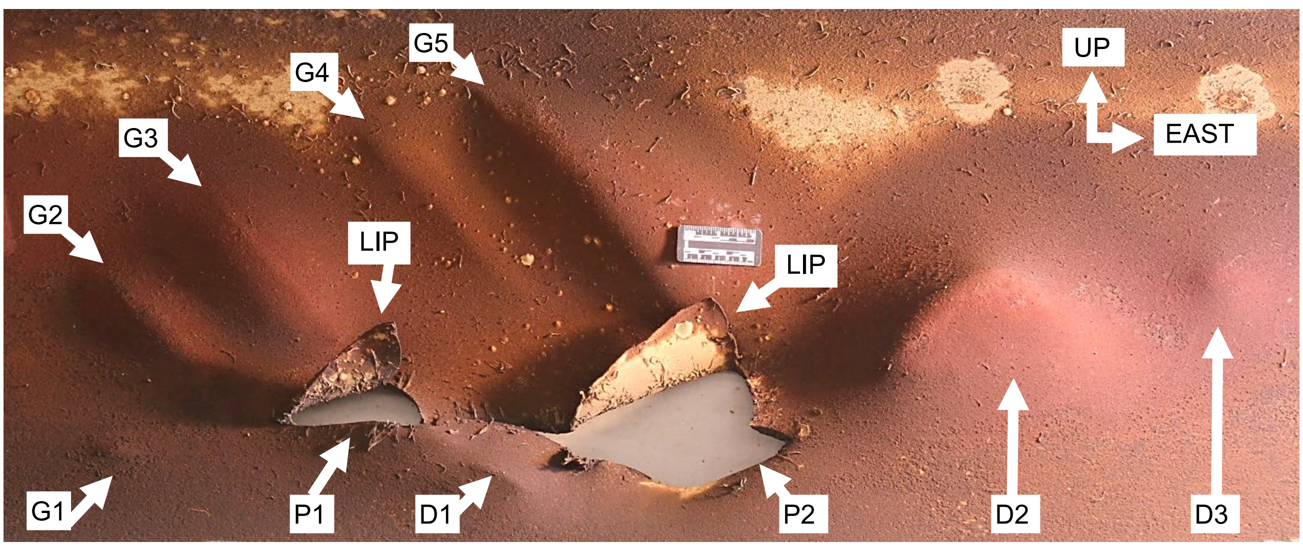


Figure 13. View of the inner face of the pipe after the rectangle portion containing the damaged portion was cut showing punctures “P1” and “P2”, dents associated with elongated dents “G1” through “G5”, and dents “D1” through “D3”. View shown prior to cleaning.

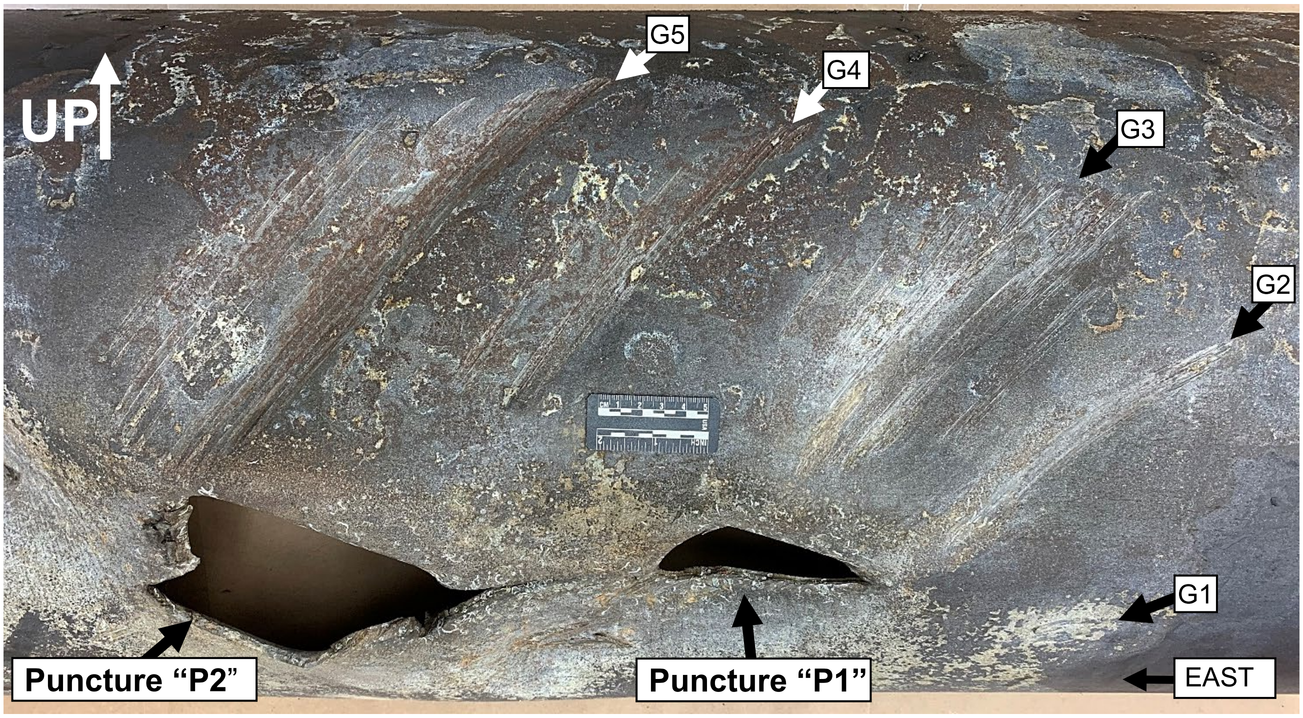


Figure 14. Outer surface of the pipe showing exposed cluster of gouges within elongated dents “G2” through “G5”, after cleaning.

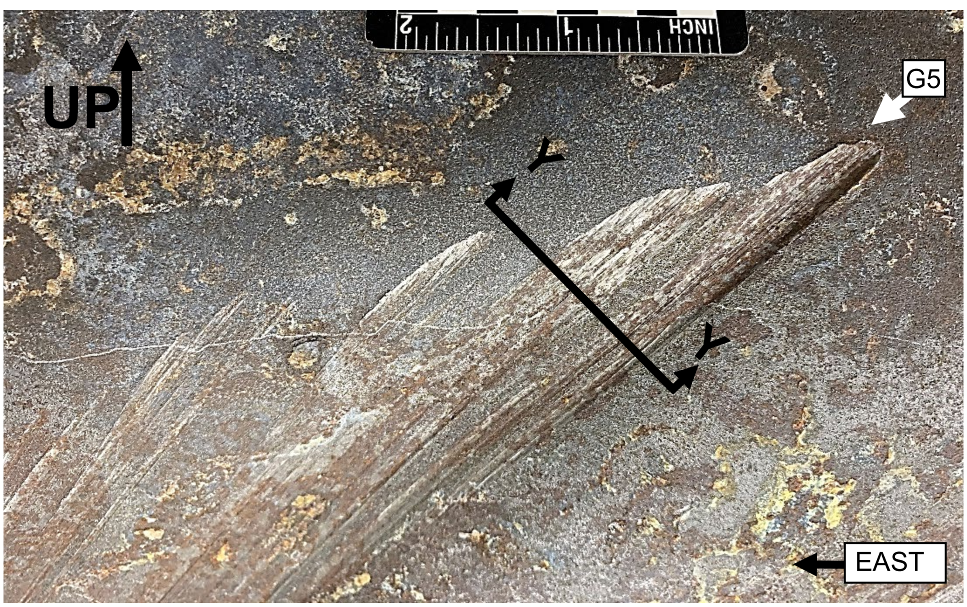


Figure 15. Close-up photograph of the outer surface of the pipe in the area of elongated dent “G5” showing several gouges at the upper end of the elongated dent, after cleaning.

Figure 16. Close-up photograph of the outer surface of the pipe in the area of dent "D1" showing two major gouges located within dent, after cleaning. Arrows indicate general direction of metal flow. Metal flow terminated in the general area indicated by a dashed line.

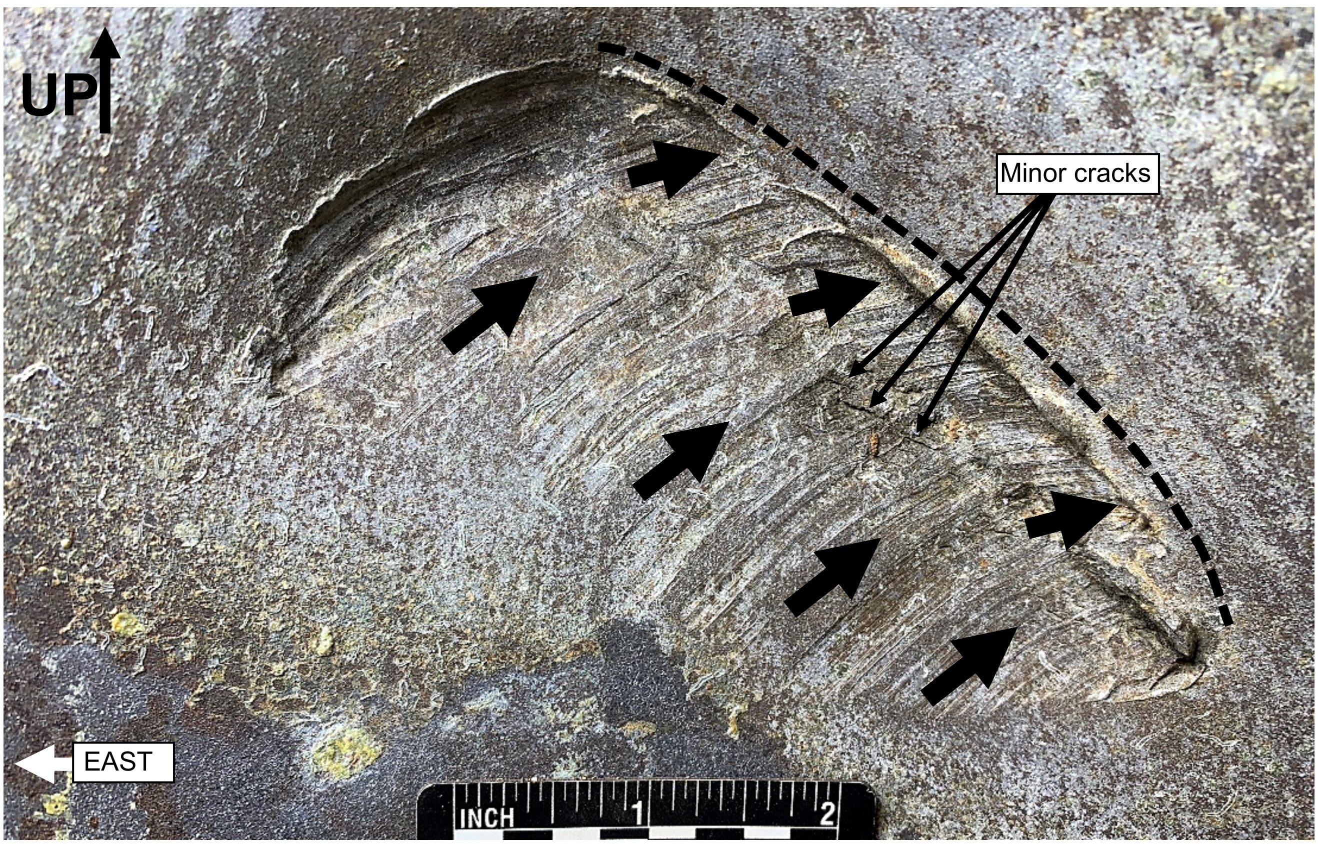


Figure 17. Close-up photograph of the outer surface of the pipe in the area of dent "D2" showing gouges within dent "D2", after cleaning. The gouges exhibited evidence of metal flow deformation that was in the general direction indicated by the arrows. Metal flow deformation terminated in the general area indicated by a dashed line (area containing metal build-up). Evidence of minor cracks were found within the dent. The cracks did not penetrate the wall.

Figure 18. Close-up photograph of the outer surface of the pipe in the area of dent “D3” showing evidence of several minor gouges within the dent, after cleaning.

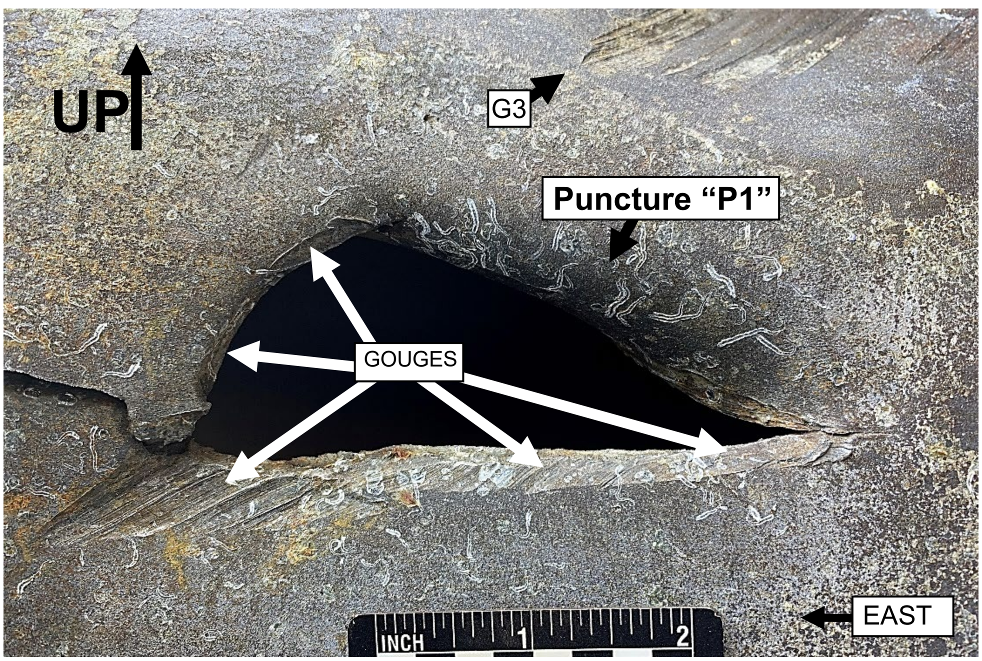


Figure 19. Close-up photograph of the outer surface of the pipe in the area of puncture “P1”, after cleaning, showing gouges along the edges of the fracture faces.

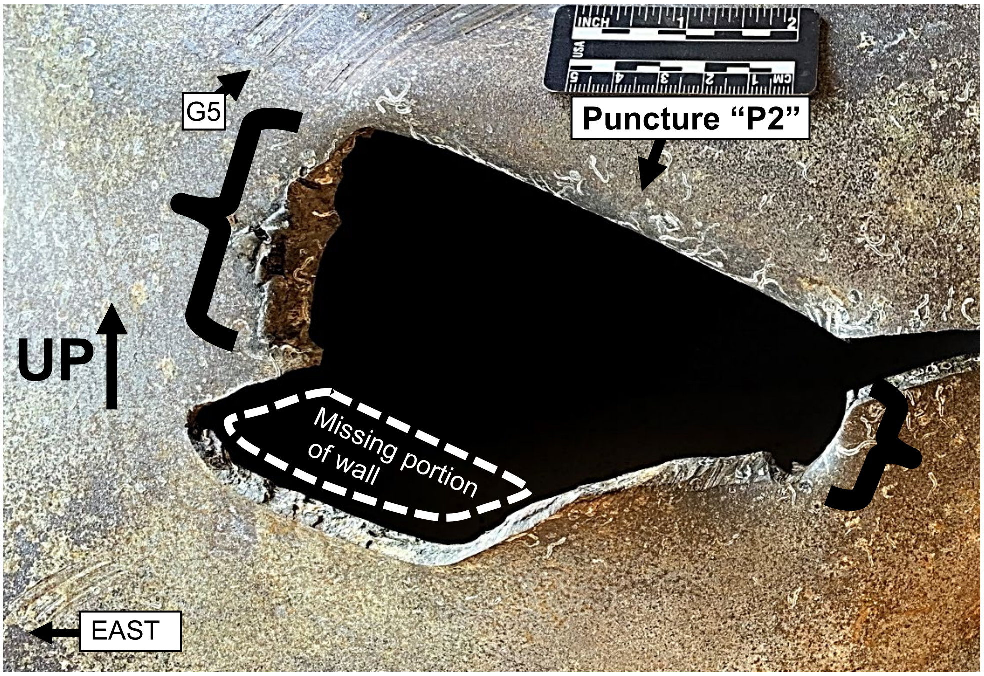


Figure 20. Close-up photograph of the outer surface of the pipe in the area of puncture "P2", after cleaning. A portion of the wall is missing from the area enclosed by a dashed line (the length of the lip portion does not extend into the missing portion of the wall). The west end of the puncture shows inward metal deformation, in the area indicated by a bracket. The east end of the puncture shows severe metal deformation, in the area indicated by a bracket.

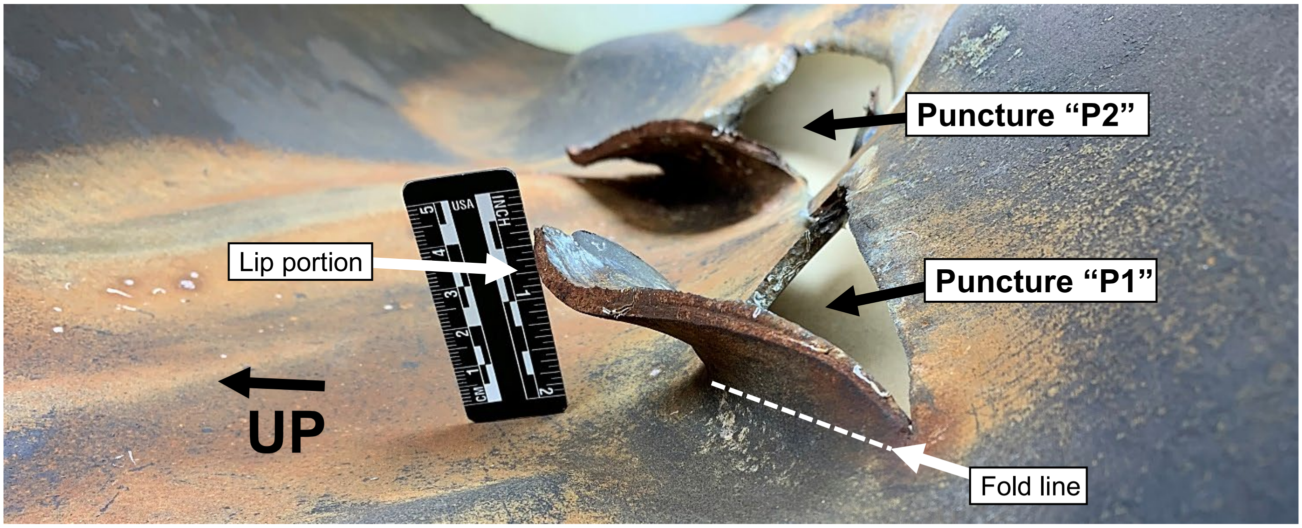


Figure 21. View looking east at the Inner surface of the pipe showing puncture "P1" in the foreground and puncture "P2" in the background, after cleaning. Dashed line indicates a fold line at the base of the lip.

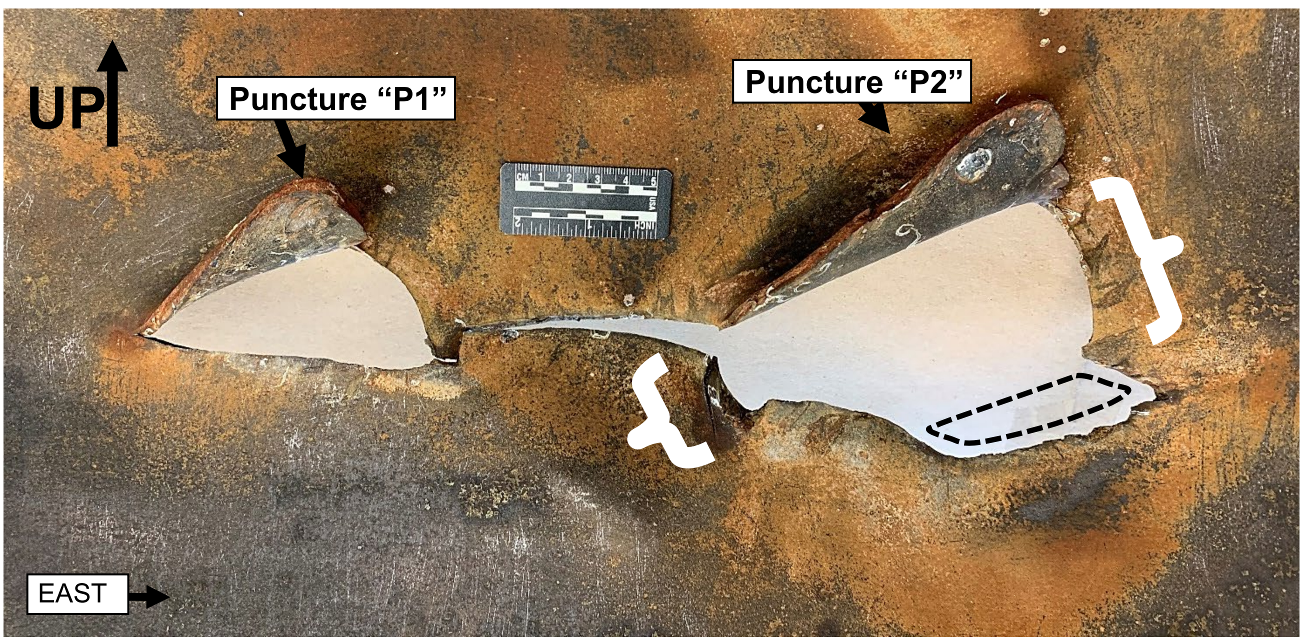


Figure 22. Inner surface of the pipe showing punctures "P1" and "P2", after cleaning. A portion of the wall is missing from the area enclosed by a dashed line. The west end of the puncture shows inward metal deformation in the area indicated by a bracket. The east end of the puncture shows severe metal deformation in the area indicated by a bracket.

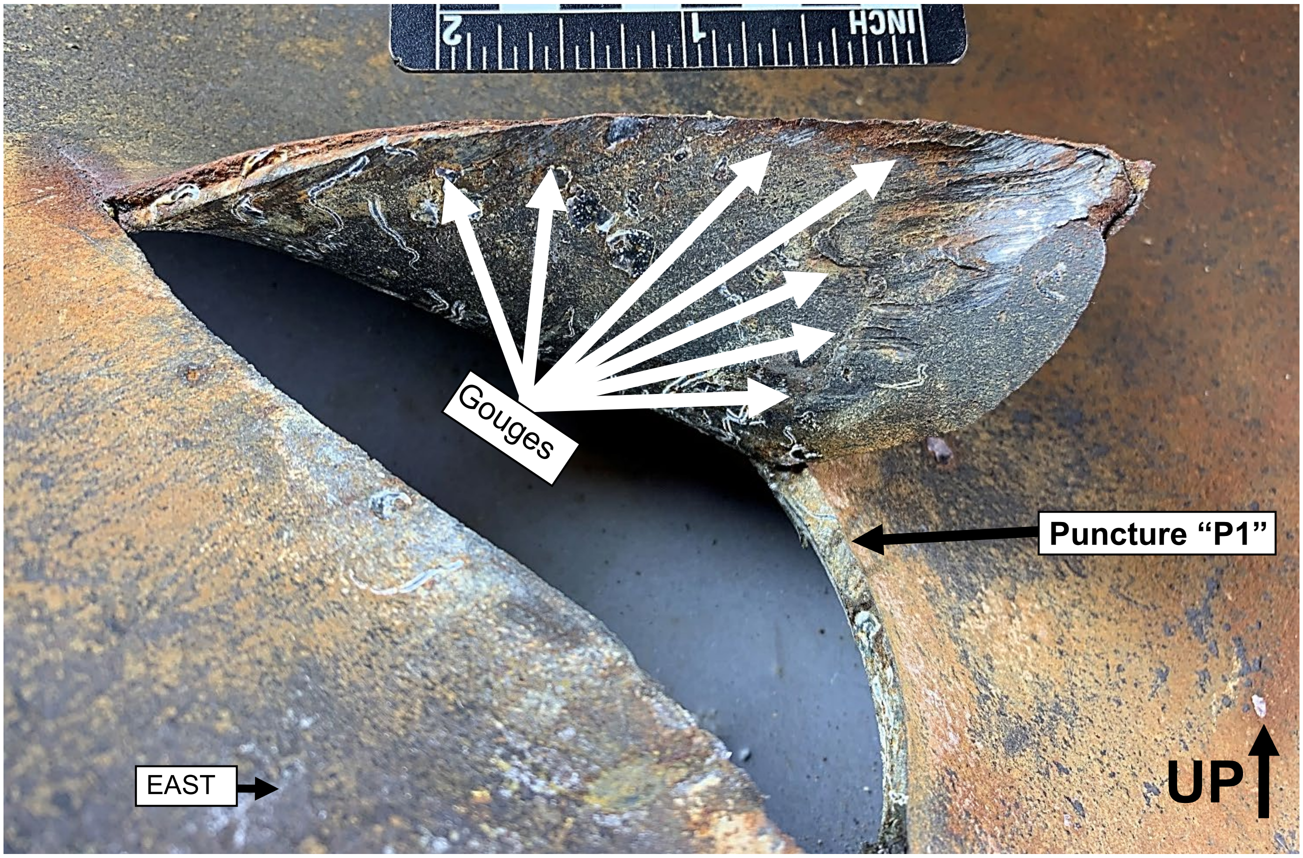


Figure 23. Close-up photograph of the inner surface of the pipe in the area of puncture "P1", after cleaning. The outer face of the lip portion at the edges of the fracture showed evidence of impact marks (deformed surface containing gouges).

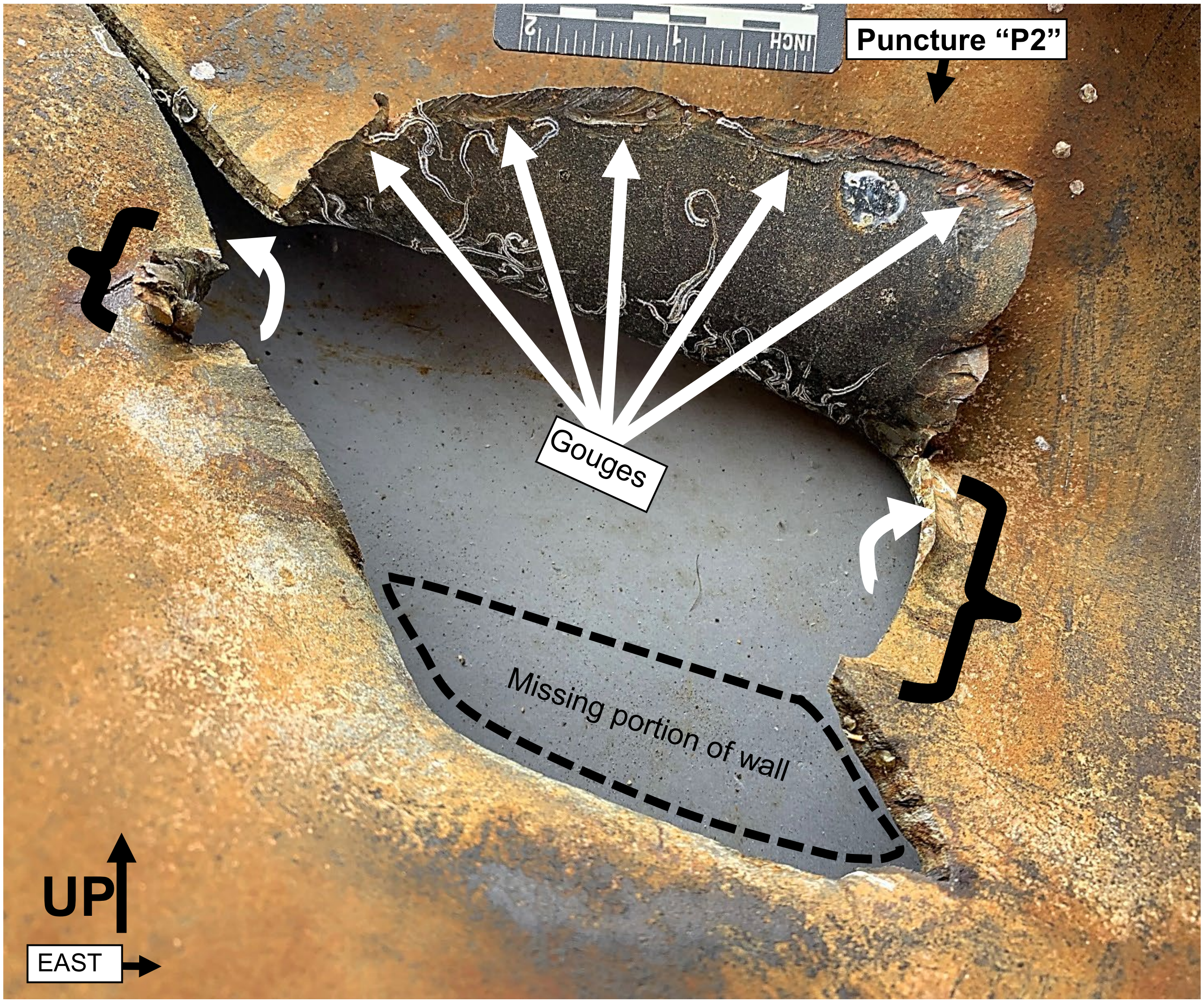


Figure 24. Close-up photograph of the inner surface of the pipe in the area of puncture "P2" after cleaning. The outer face of the lip portion at the edges of the fracture showed evidence of impact marks (deformed surface containing gouges). The wall of the pipe at the east and west ends of the puncture, in the areas indicated by brackets, showed inward bending deformation (in the general direction indicated by arrows). A portion of the wall is missing from the area enclosed by a dashed line.

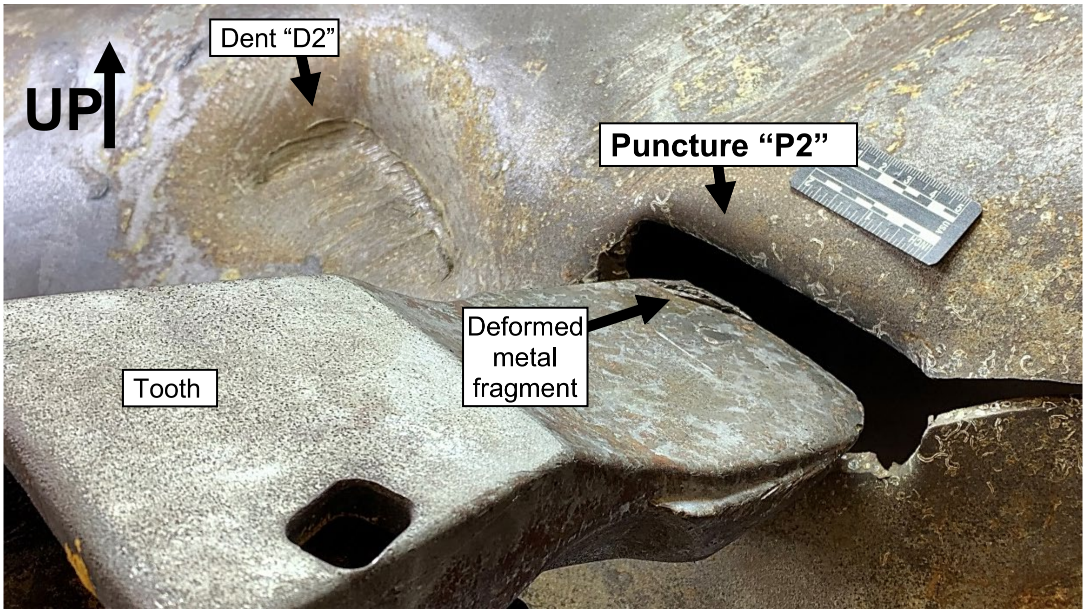


Figure 25. Oblique view of puncture "P2" with the tip portion of tooth "C1" facing the puncture and dent "D3" containing gouges in the background, after cleaning. The tip of tooth "C1" contained evidence of a deformed metal fragment.

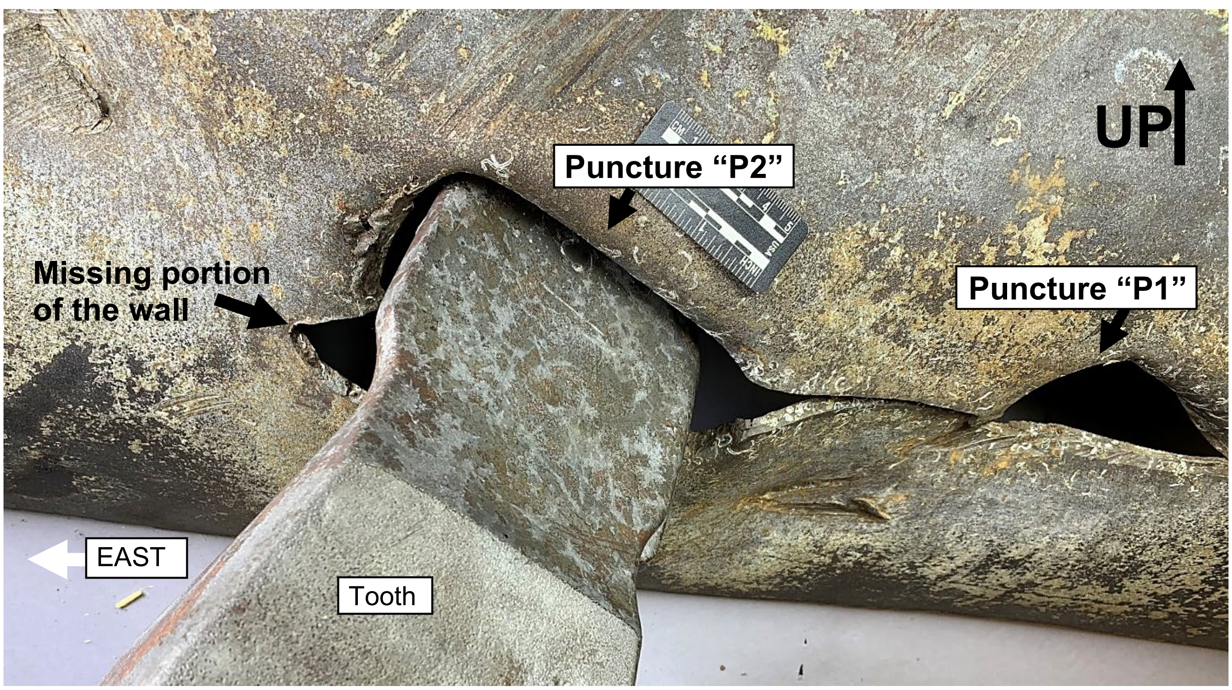


Figure 26. Oblique view of puncture "P2" with the tip portion of tooth "C1" partially inserted into the puncture, after cleaning.

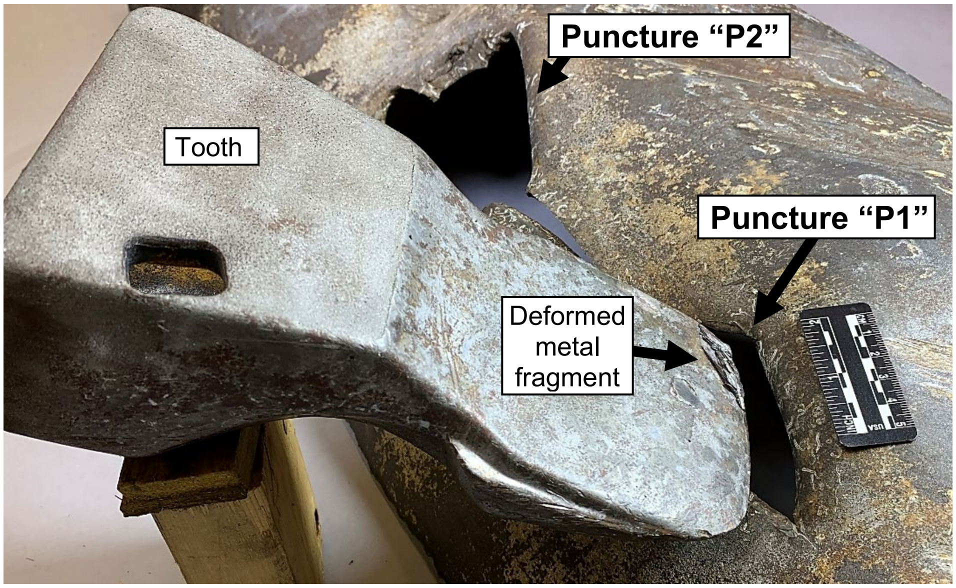


Figure 27. Oblique view of puncture “P1” with the tip portion of tooth “C1” facing the puncture, after cleaning. The tip of tooth “C1” contained evidence of a deformed metal fragment.

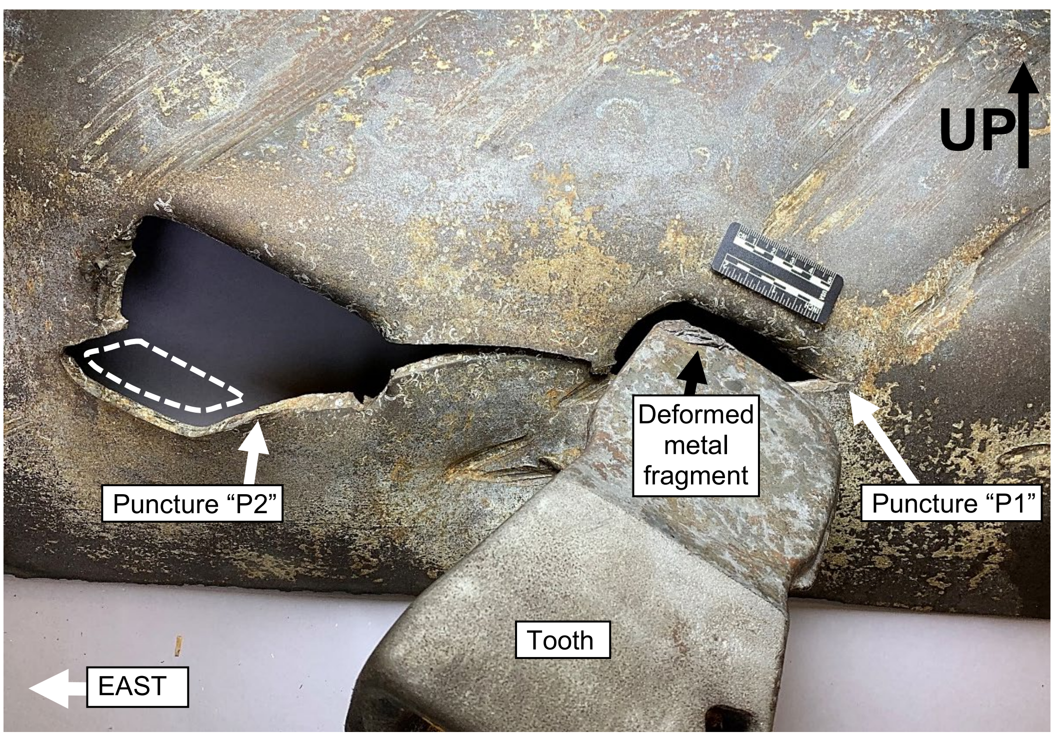


Figure 28. Oblique view of puncture “P1” with the tip portion of tooth “C1” facing the puncture, after cleaning. The tip of tooth “C1” contained evidence of a deformed metal fragment. A portion of the wall is missing from the area enclosed by a dashed line.

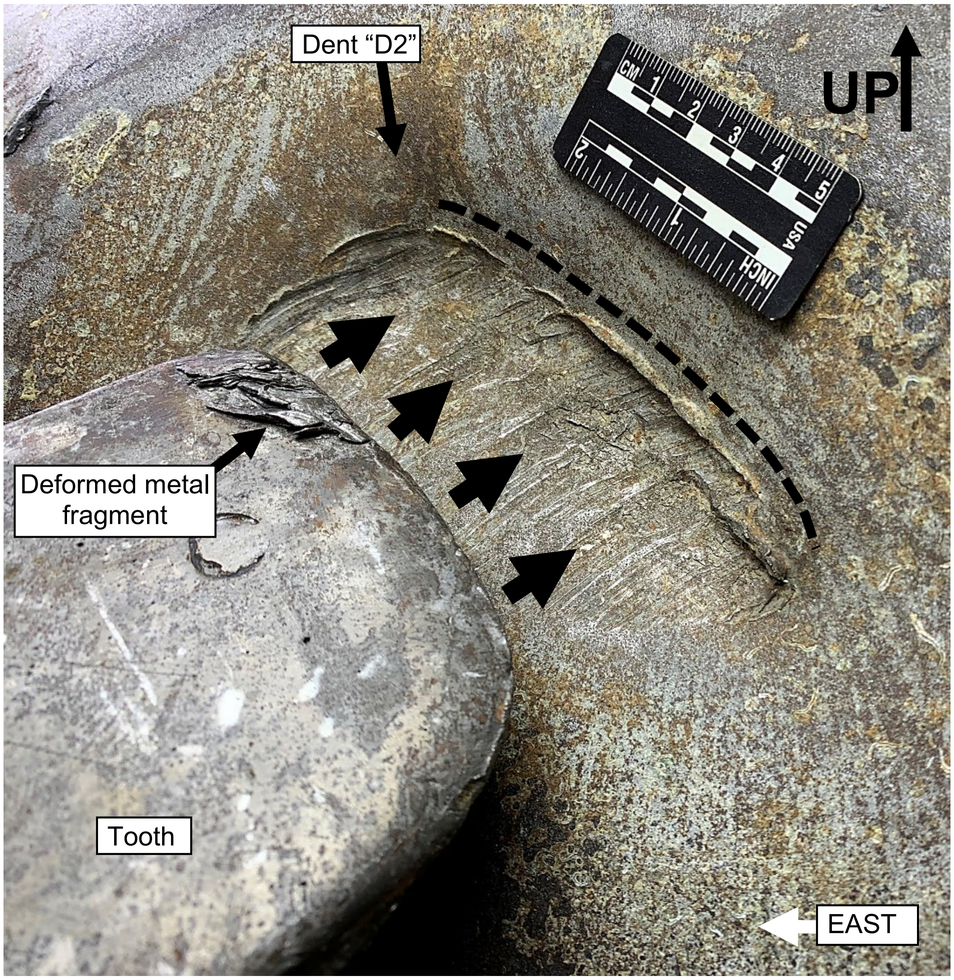


Figure 29. Outer surface of the pipe in the area of dent "D2" with tooth "C1" positioned next to the dent, after cleaning. The dent contained gouges consistent with metal flow in the general direction indicated by black arrows. Metal flow deformation terminated in the general area indicated by a dashed line. The tip of tooth "C1" contained evidence of a deformed metal fragment.

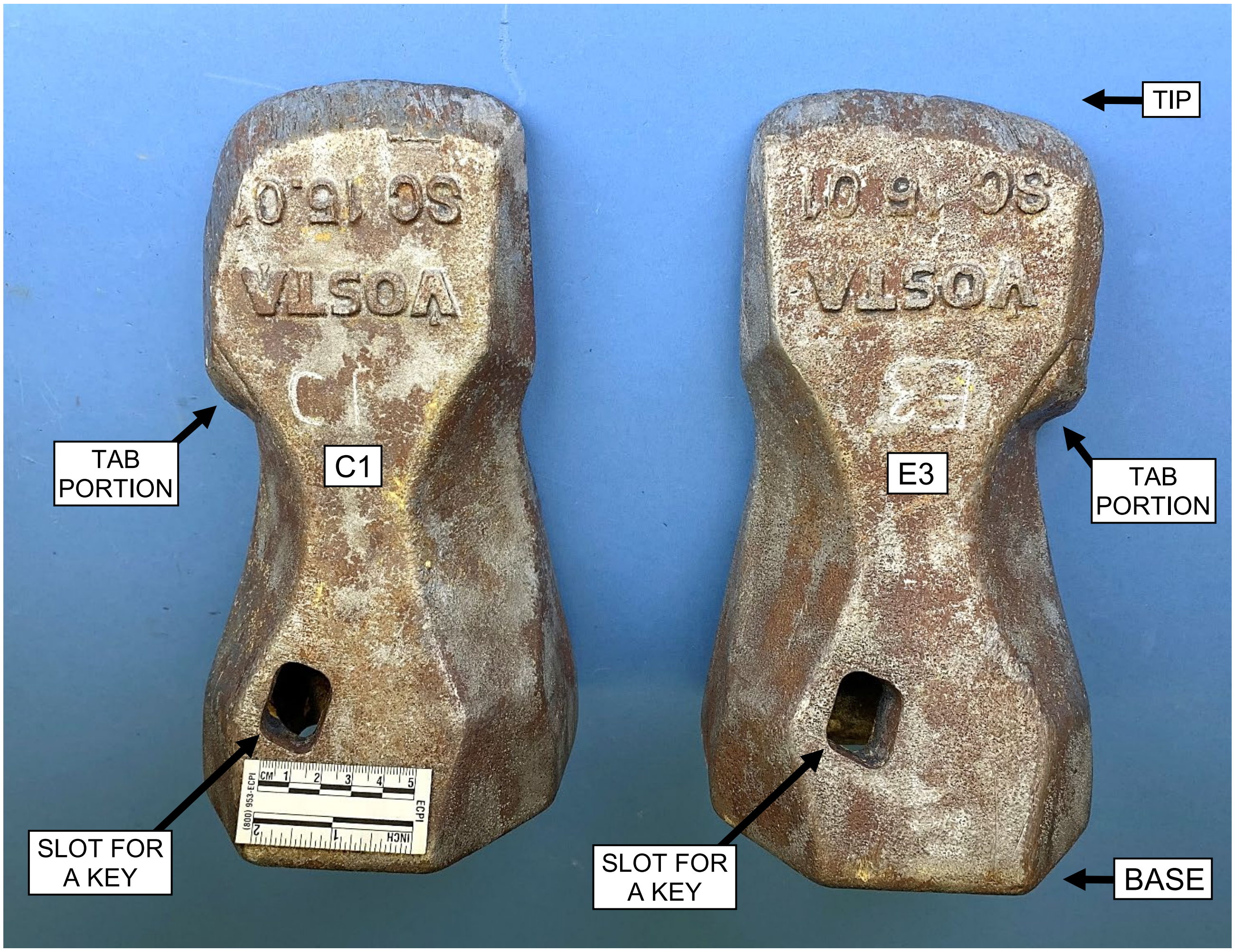


Figure 30. Front face of teeth "C1" and "E3", respectively, after cleaning. The slot for the key for each tooth is located at the base portion on the left side. On each tooth, the slot for the key is located on the same respective side, but the tab portion is located on opposite respective sides.

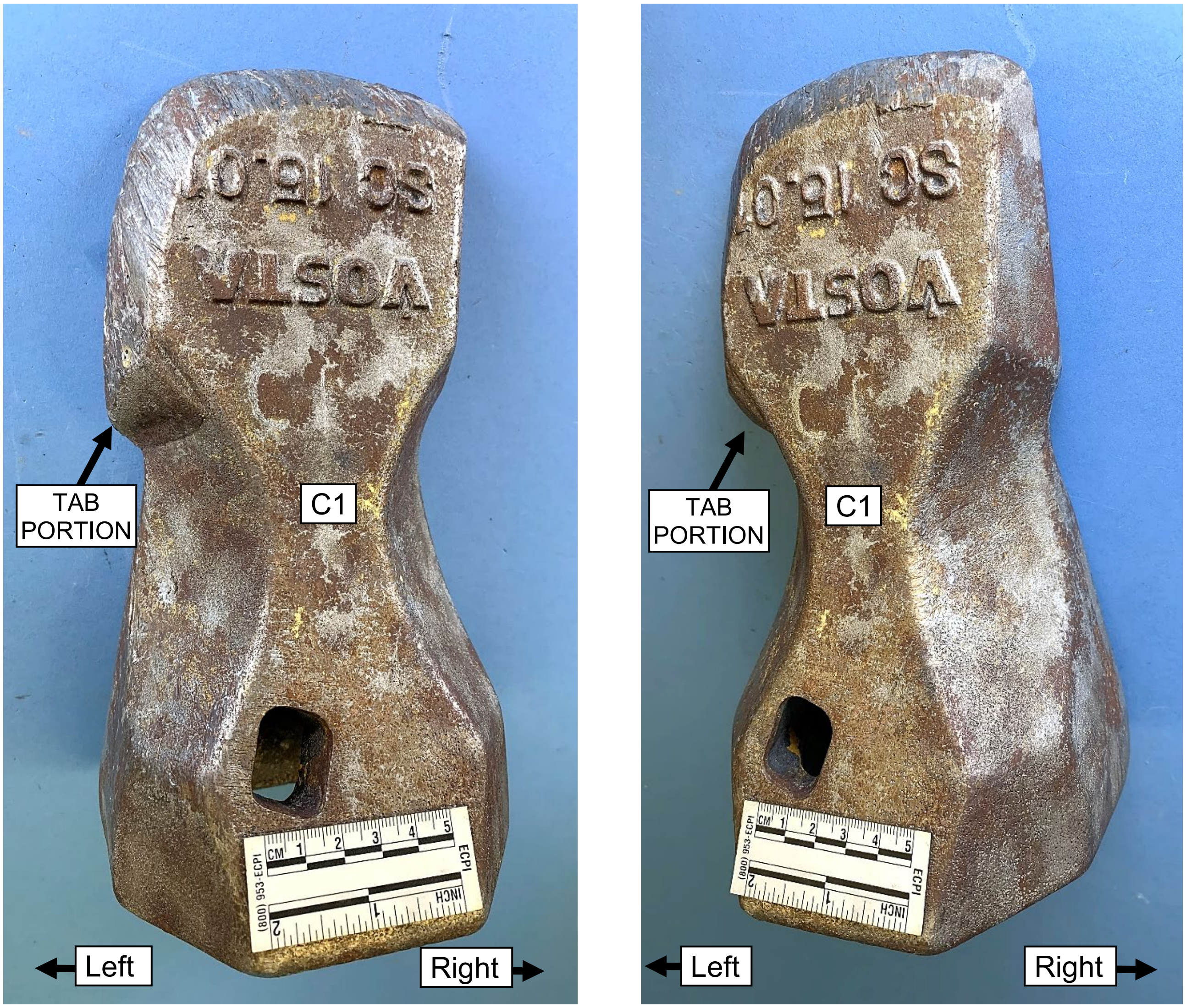


Figure 31. Front face of tooth “C1” showing the left and right edges, respectively, after cleaning.

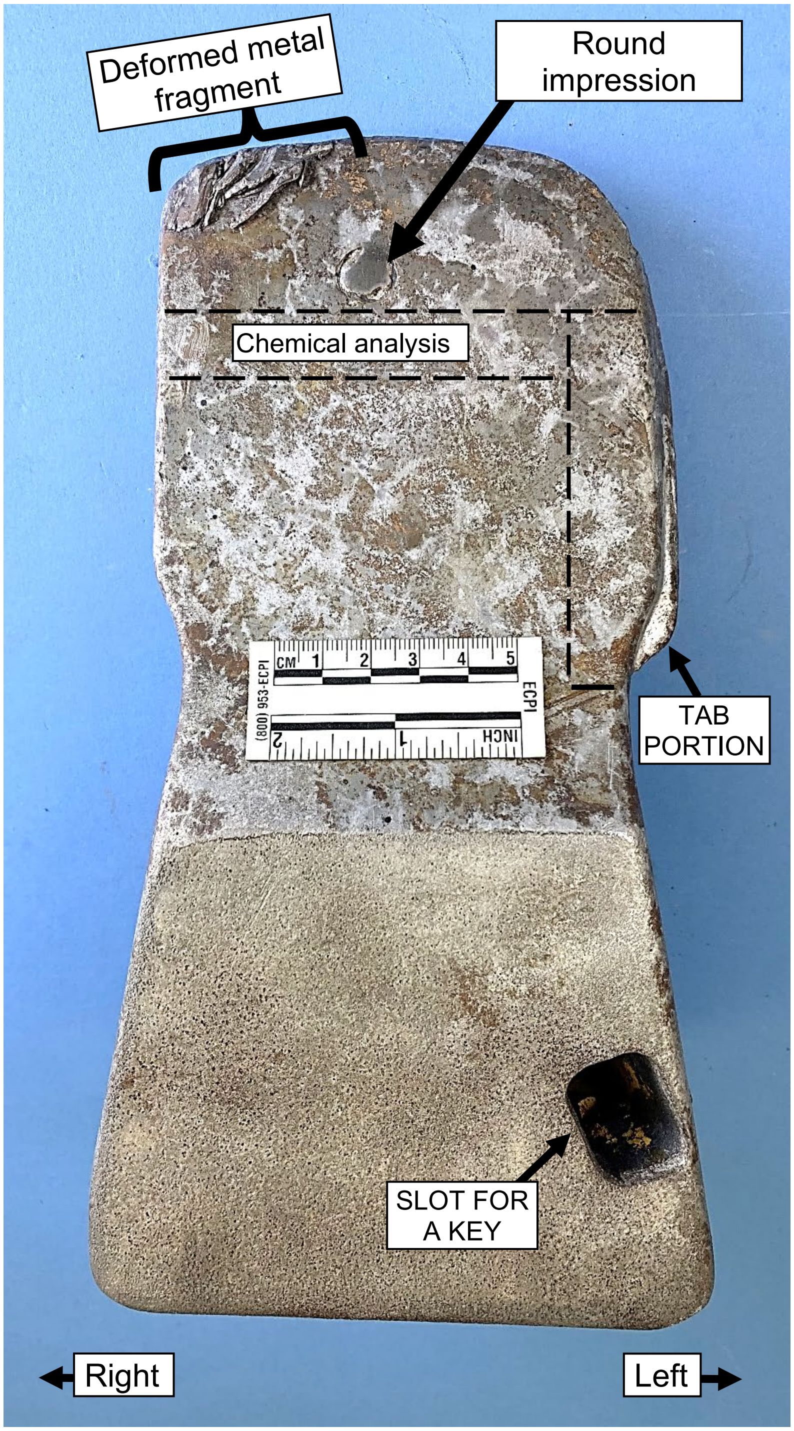


Figure 32. Back face of tooth "C1" after cleaning, showing a deformed metal fragment that was imbedded at the tip portion. Dashed lines indicate cuts made to facilitate examination of the gouges at the tip of the tooth and for chemical analysis.

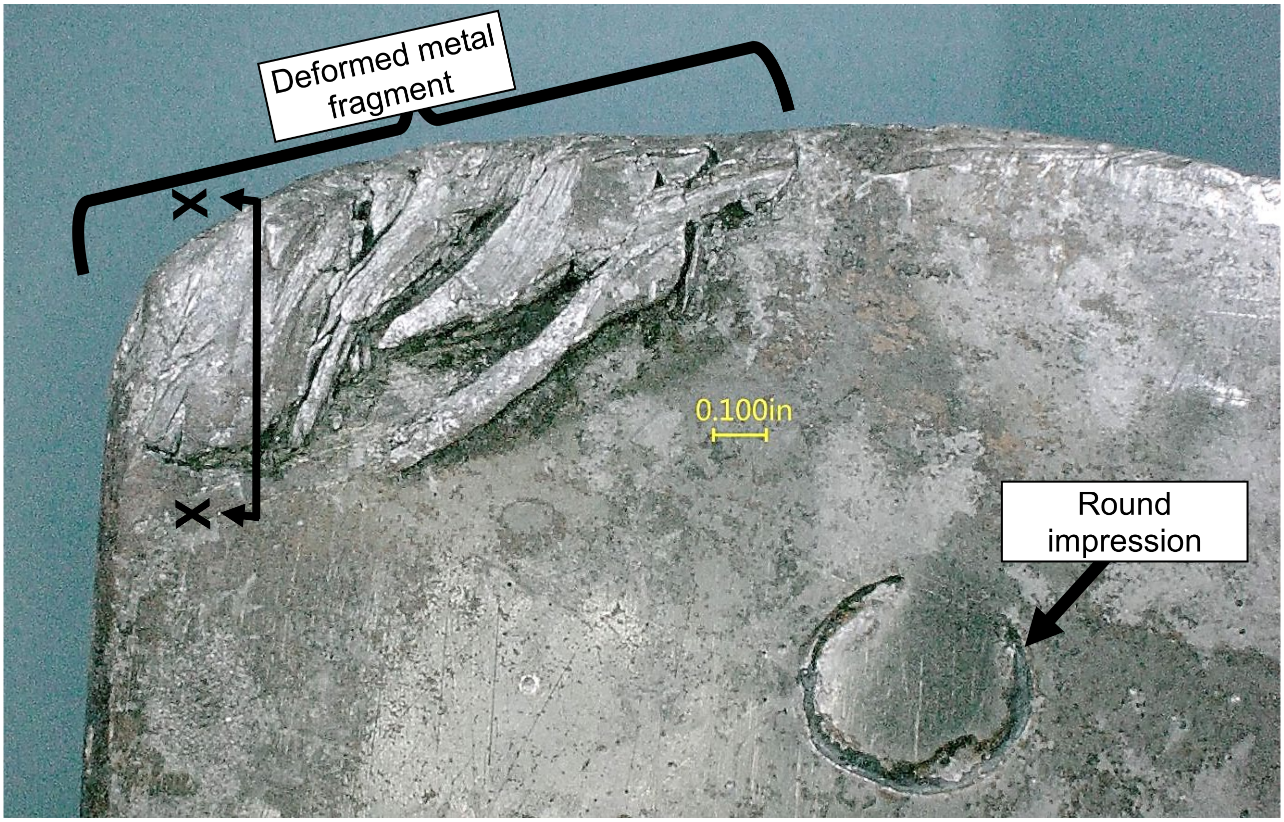


Figure 33. Back face of tooth “C1” after cleaning showing deformed metal fragment that was imbedded at the tip portion.

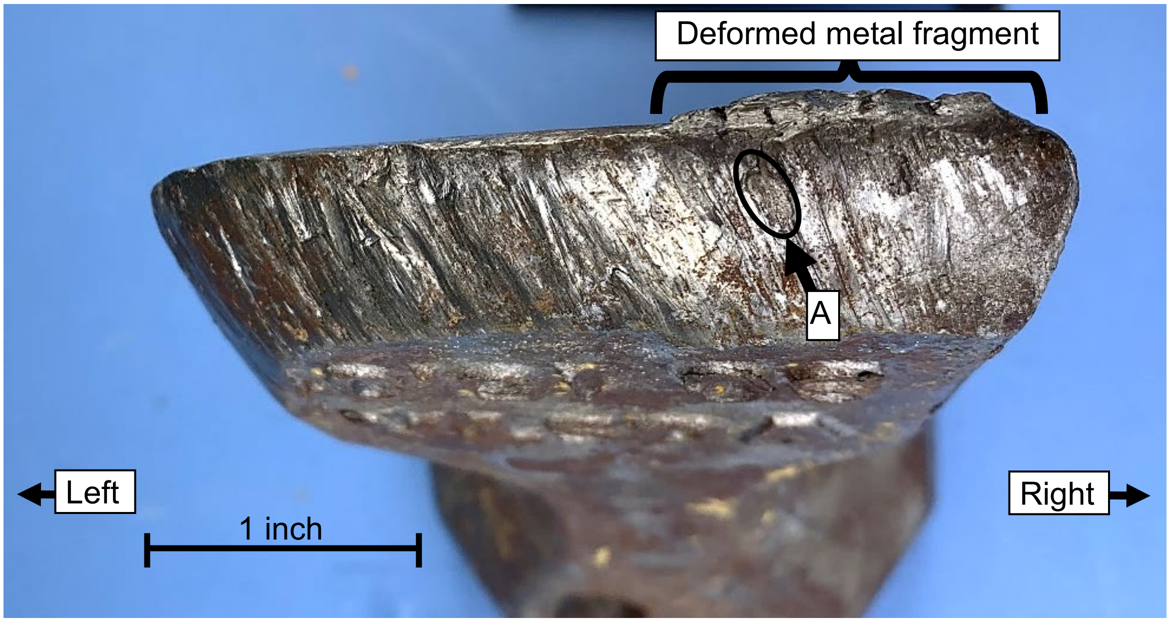


Figure 34. View of the tip portion of tooth “C1” showing deformed metal fragment at the back face, after cleaning. The abraded surface at the tip of the tooth also showed evidence of metal build-up in the area indicated by arrow “A”.

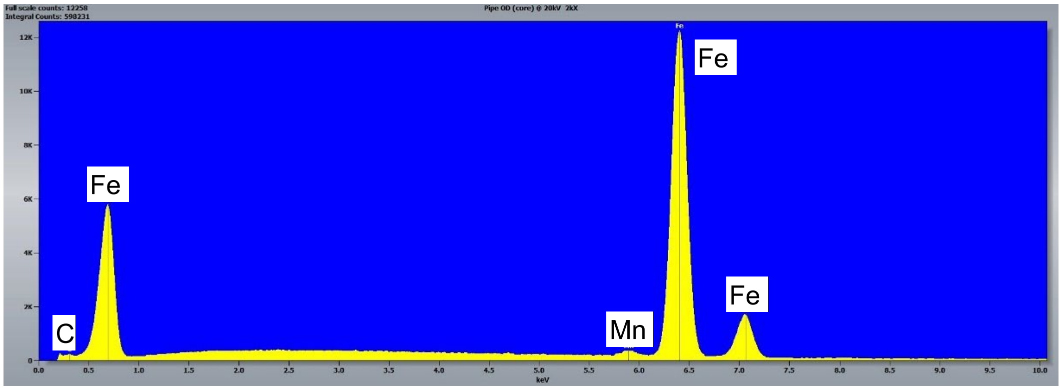


Figure 35. EDS spectrum of the core portion of the pipe wall.

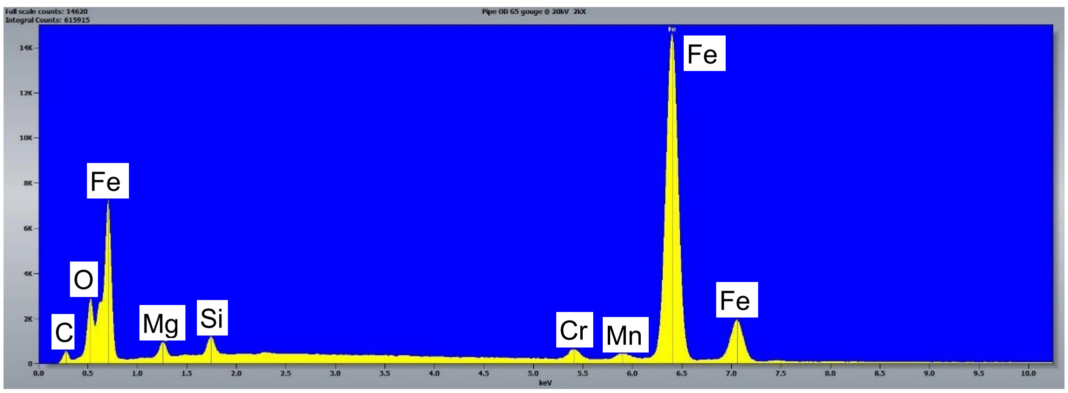


Figure 36. EDS spectrum of gouges at the upper end of elongated dent "G5".

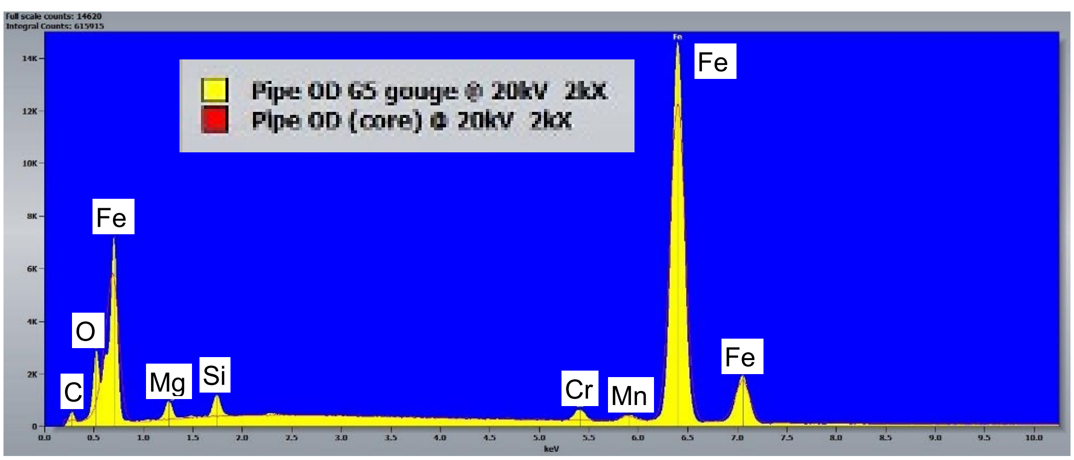


Figure 37. EDS analysis of the core portion of the pipe (red outline) compared with the spectrum from the gouges at the upper end of elongated dent "G5" (solid yellow).

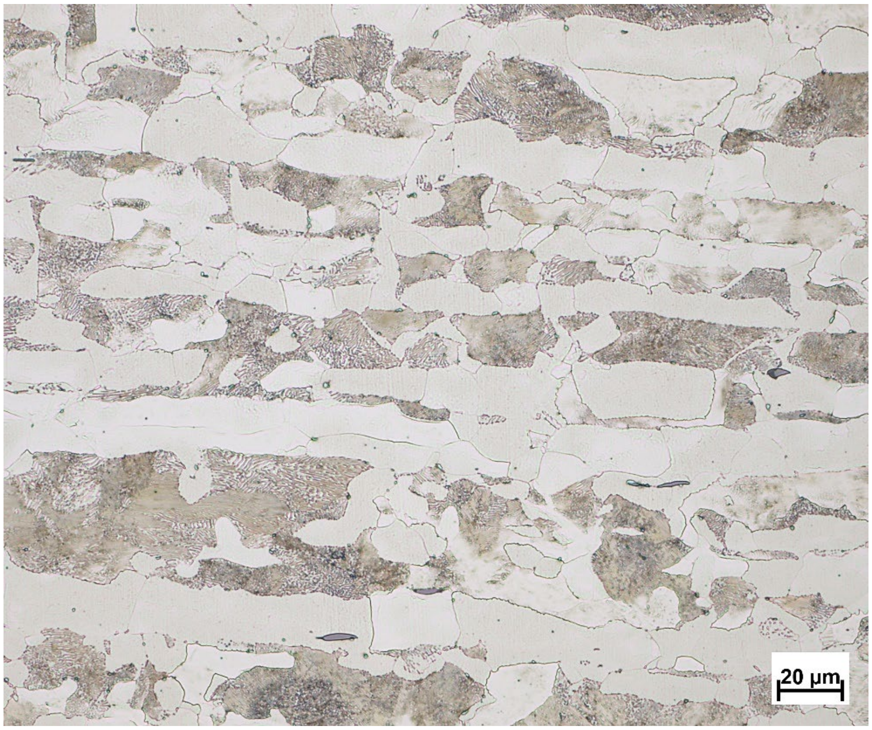


Figure 38. Microstructure of the pipe wall at the core showing banded pearlite and ferrite grains. Etched with 2% Nital reagent. 500X

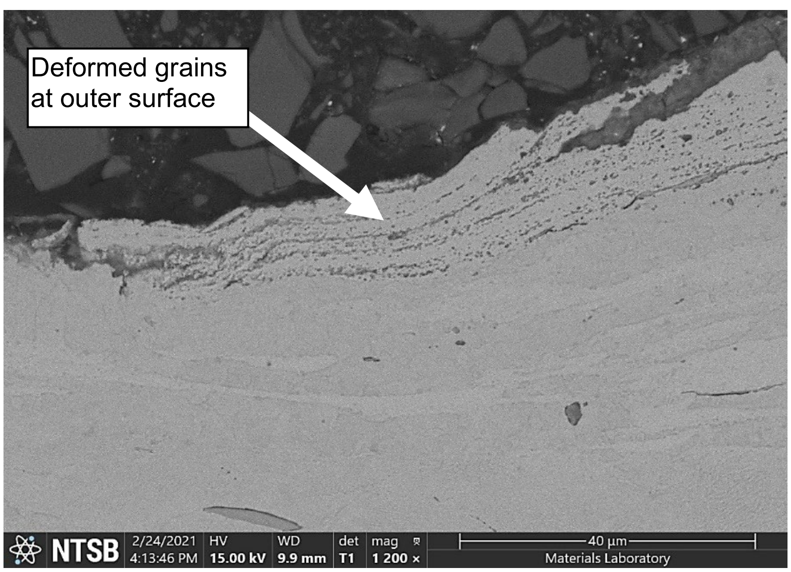


Figure 39. SEM image of the pipe wall cross section showing severely deformed grains at the outer surface. Etched with 2% Nital reagent. 1,200X

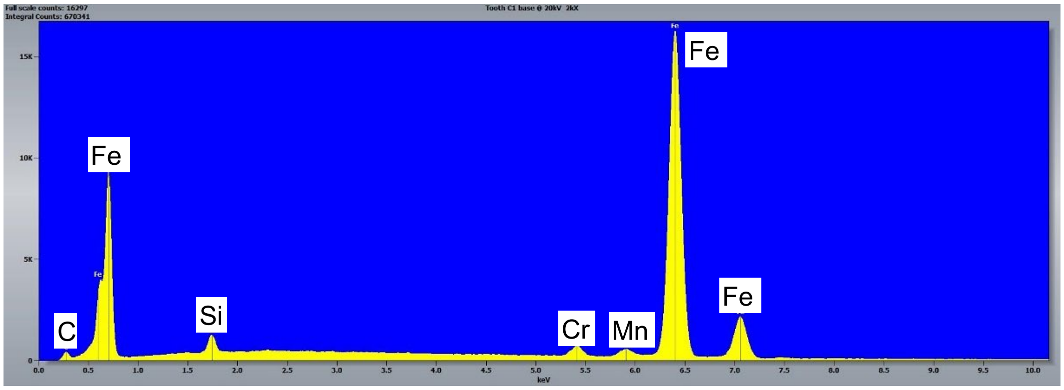


Figure 40. EDS spectrum of the core portion of tooth “C1”.

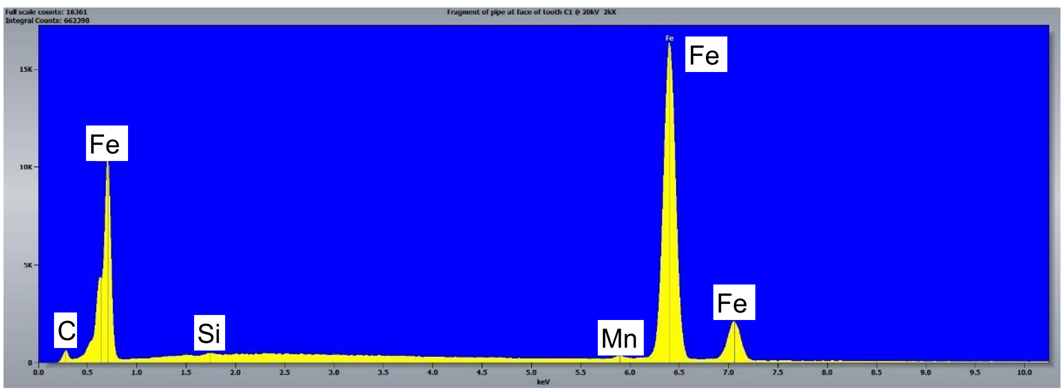


Figure 41. EDS spectrum of the deformed metal fragment.

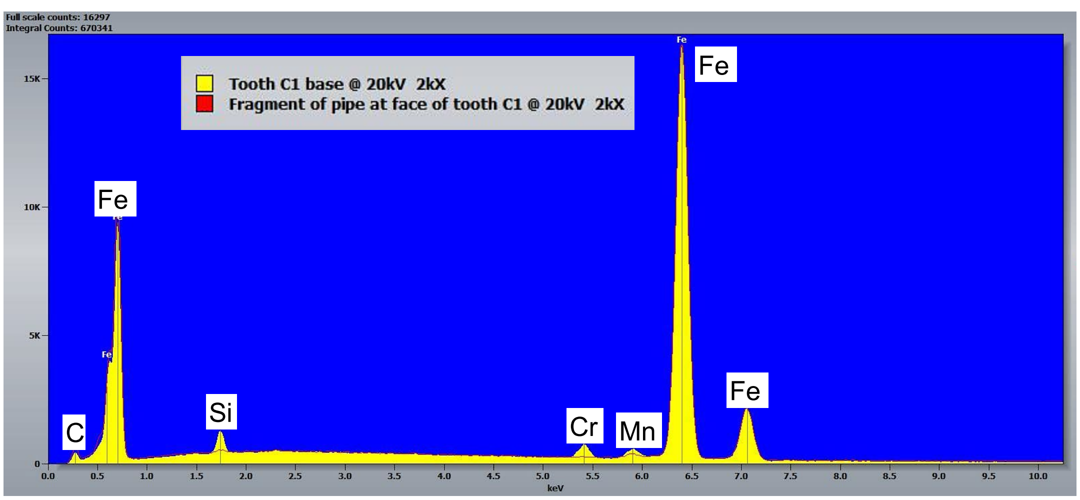


Figure 42. EDS analysis of the core portion of tooth “C1” (solid yellow) compared with the spectrum of the deformed metal fragment (red outline).

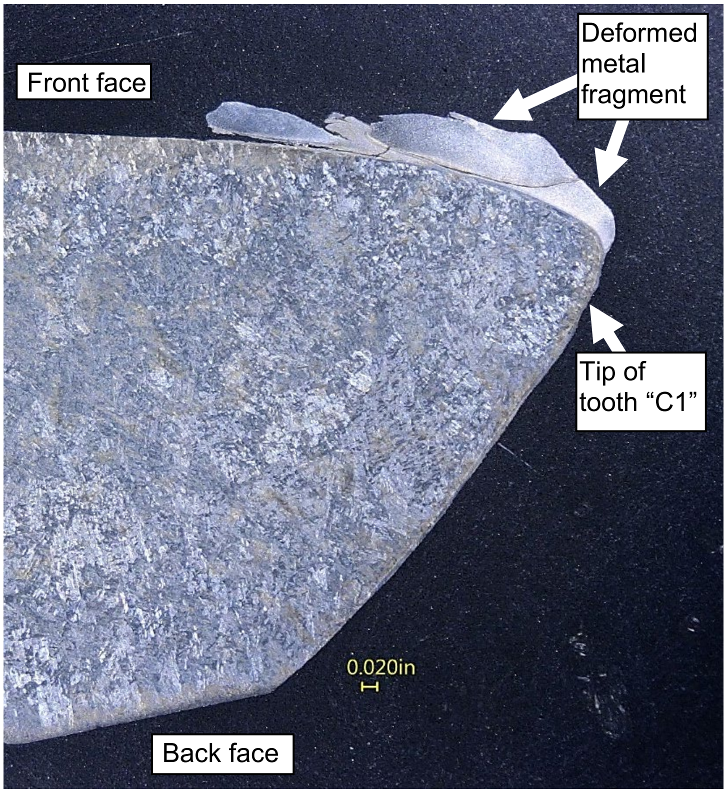


Figure 43. Metallurgical section “X-X” in figure 33 showing the tip of tooth “C1” and deformed metal fragment. Etched with 2% Nital reagent.

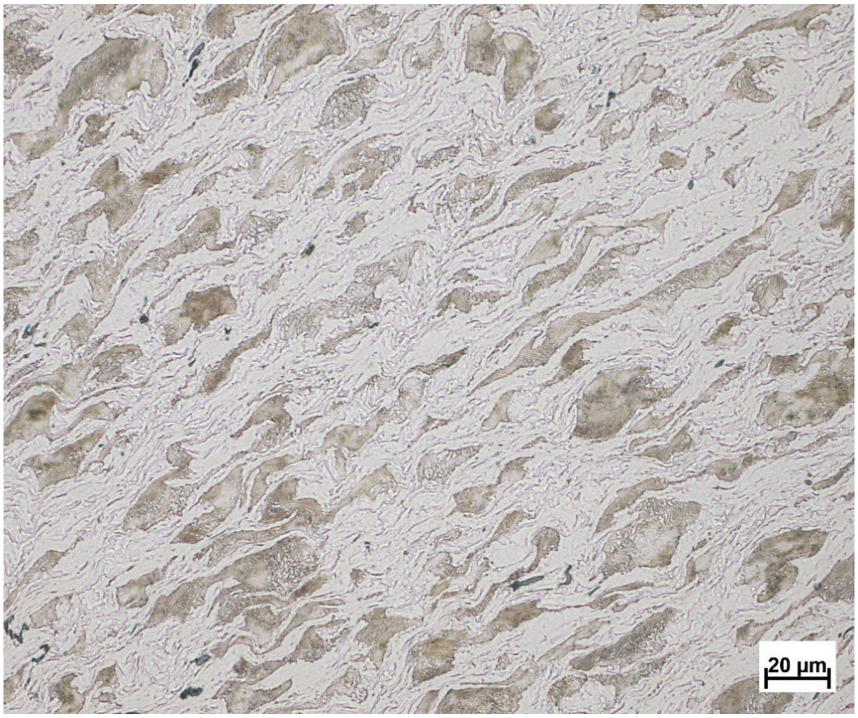


Figure 44. Microstructure of the deformed metal fragment at the tip of tooth “C1” showing pearlite and ferrite grains that exhibited evidence of deformation. Etched with 2% Nital reagent. 500X

Figure 45. Microstructure of the tooth “C1” at the core showing a cast microstructure. Etched with 2% Nital reagent. 500X

APPENDIX 1

NTSB PIPE RECOVERY PROTOCOL

(5 Pages)

National Transportation Safety Board
Corpus Christi, Texas
NTSB Accident DCA20FM026

Protocol for Recovery, Crating, and Shipping Damaged Pipe to NTSB

This protocol addresses recovery of a damaged 16-inch diameter pipe that is submerged underwater. Mechanical damage (punctures, gouges, and deformation associated with the punctures) was reported in the area "East" of the girth weld (GW), as indicated in the attached drawing. Recovery team will remove the following:

> Segment of pipe containing mechanical damage on the exposed metal surface (portion between 2 feet west of the GW and 12 feet 4-inches east of the GW). This can change based on follow-up onshore visual examination.

NTSB personnel will not be present on-site but will monitor the activity remotely. Four "checkpoints" are identified in this document; they indicate critical phases of the operation where information must be relayed to the investigator-in-charge (IIC) Luke Wisniewski and the Pipeline Group Chairman Paul Stancil, so that NTSB can make decisions where to cut the pipe and ship the pieces.

NTSB acknowledges that Enterprise Products Operating LLC operates the propane pipeline (Line TX219) that is the subject of the NTSB investigation and that has provided the NTSB and other parties with the Enterprise Procedure for Removal of Breached Piping from Line TX219 (the Enterprise Procedure). Enterprise's contractors will execute the work in accordance with the Enterprise Procedure. Nothing in this protocol is intended to abrogate Enterprise's property rights with respect to TX219, including the segment of interest.

Underwater Recovery

The Enterprise Procedure describes the specific process for the underwater recovery of the piping segment. Care shall be taken to preserve the exposed metal portion of the pipe that contains mechanical damage (fracture surfaces, depressions [dents], and gouges). Handle the pipe with equipment/slings that do not introduce additional damage to the pipe.

Record the GPS location of the GW, cut ends, and center of each hole.

Using a suitable underwater marking method, place marks on the submerged pipe indicating orientation. At minimum, mark the following on the pipe: top dead center (TDC), east and west end, and use arrow to indicate product flow direction. At a later time, the pipe can be marked more completely in a permanent manner.

In preparation for the removal of the pipe segment, Enterprise will instruct the divers to watch for cutter tooth fragments. The fractured tooth may be located at the inner surface of the pipe or at the sea floor below the pipe If observed, the divers will attempt to recover the fragments in a secure manner, such as in a plastic bag, for later examination on shore. Enterprise, however, is not committing to undertake a systematic search for any potential evidence beyond the recovery of the pipe segment of interest

Pursuant to the Enterprise Procedure, Enterprise Products will recover a 14-foot 4-inch long pipe segment that includes the GW. Cuts will be made 2 feet west of the GW and 12 feet 4 inches east of the GW. The cut at the east end will be done in an area that has coating all around the pipe. Enterprise may determine that a longer segment is necessary. Cuts can be made by mechanical method, such as with a saw. Concrete coating can be removed as necessary from areas to be cut by mechanical equipment.

Once the pipe segment is on the recovery vessel/barge, lay down the pipe on a plastic tarp or wood blocks. Do not allow the recovered pipe segment to be dragged on the deck.

With a hose wash the outer and inner surface of the pipe with abundance of fresh water. Use compressed air to dry the pipe.

On-shore

NTSB acknowledges that Enterprise will provide the NTSB and other parties with the Enterprise's procedure for preservation, scanning, inspection, and transportation. Enterprise's contractors will execute the work detailed therein in accordance with that procedure.

The pipe will be moved to (**fill in location details**), a secure facility that has access to tools and good lighting, and for preliminary visual examination of the pipe.

Visually examine the outer surface of the pipe. According to the drawing, two depressions (dents) are located at 3 o'clock. The larger dent is on the east side. Examine the pipe for evidence of mechanical damage on the exposed metal surface in areas that extend beyond the eastern edge of the larger dent (look for mechanical damage that was not reported on the drawing). Determine if such gouge or dent is on the exposed metal surface.

The recovered pipe portion will be scanned by a 3D scanner (details on process and equipment to be provided). Reflective markers can be temporarily placed on pipe for reference points, provided that they are not placed over dent or gouge areas. Replicas of damaged areas, such as silicone casting, will not be permitted.

At this point, parties to NTSB's investigation may visually inspect, measure, and photo-document the recovered pipe portion All data collected will be shared with NTSB and parties to NTSB's investigation.

Checkpoint 1:

- Send the following information to the IIC and Pipeline Group Chairman for further assessment:
  - GPS location of the GW, cut ends, and center of each hole.
  - Provide sufficient photographs of the entire recovered pipe length that shows the mechanical damage.
  - If new damage is found (beyond what was identified on the attached drawing), make a chalk mark at the eastern most edge of the damage (dent or gouge) and send close-up photographs of the newly discovered damage.
  - Document and provide the measured distances of new damage relative to the larger dent and relative to the west GW.
  - Provide the total length of the cut portion of pipe recovered; and distance each cut end is located relative to the GW.
  - Provide 3D scan file.
  - Based on observations, the IIC and Pipeline Group Chairman can determine whether additional adjustments are necessary.

Apply liberal amount of WD-40 to the exposed metal surfaces. Soak a rag or sponge with WD-40. Manually reach through each hole and coat the inner surface in the area of the hole with liberal amount of WD-40. The hole portions may contain sharp edges. Perform this procedure with due care so as not introduce damage to the pipe and take precautionary steps to prevent injury to any exposed skin on the arm or hand.

Wood Crate

The pipe shall be shipped inside of a wood crate. The crate shall be constructed in a manner that will secure the pipe inside the crate and prevent movement of the pipe.

The crate shall be constructed in a manner such that wall of the crate shall not extend more than 6 inches beyond each cut end of the pipe, to minimize the length of the constructed crate.

Prior to placing the pipe in the crate, re-apply WD-40 to the exposed metal surfaces.

Shipping

The weight and length of the crate with pipe will dictate the shipping destination. The critical factor is the weight. The NTSB Materials Laboratory at the DC facility can accept shipment that does not exceed 3,000 pounds (limit capacity of forklift).

Prior to shipping to NTSB, weigh the crate with the pipe. This can be done with a fish-hook type scale or taking the truck to a weigh station (weigh with and without the crate).

Checkpoint 2:

- Report weight and dimensions of the crate to the IIC and Pipeline Group Chairman.
- The IIC and Pipeline Group Chairman will return with a response providing instruction where to ship the pipe.

Checkpoint 3:

- A representative from the Coast Guard will assure that a chain of custody form is filled out and will attach a tamper-indicating seal (provided by the Coast Guard) to the crate.

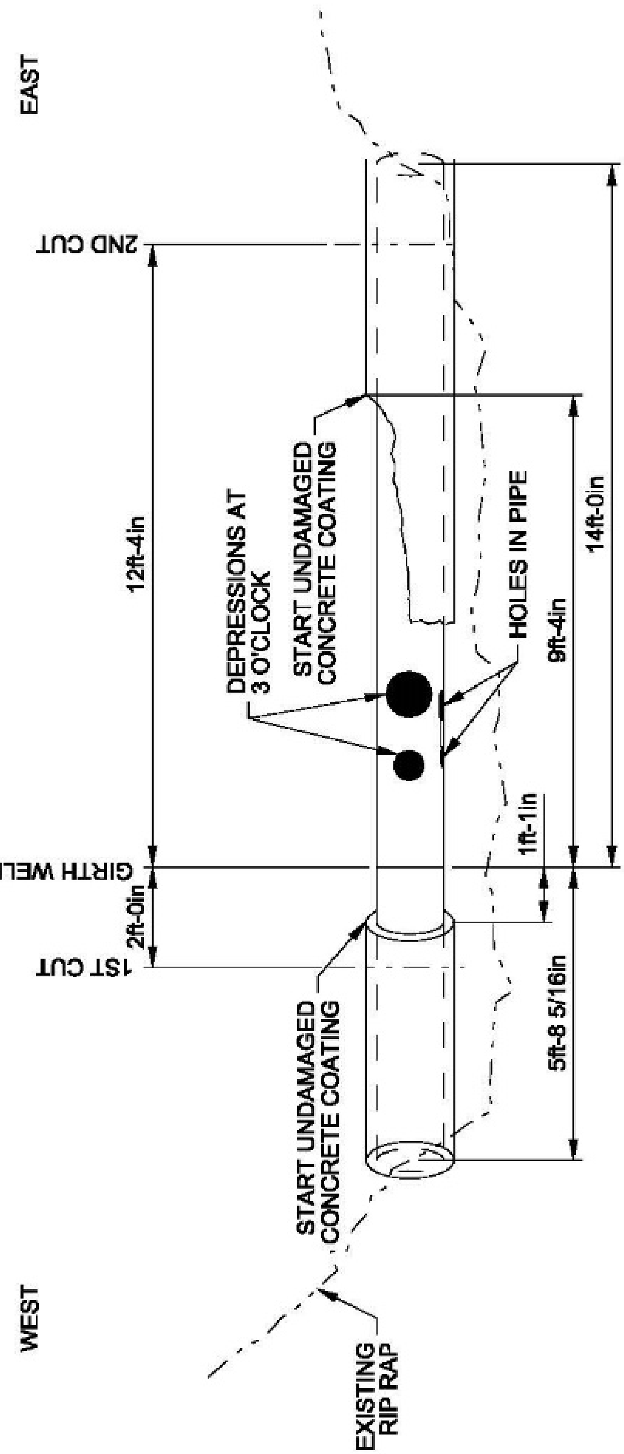
If crate is shipped on an open flat-bed truck, the crate shall be covered with a waterproof tarp. Shipment to be made with bonded trucking firm that handles chain of custody shipment.

Checkpoint 4:

- Notify the IIC and Pipeline Group Chairman when truck departs site and approximate time of arrival.

DRAWING OF PIPE SHOWING DAMAGED AREA AND LOCATION FOR CUTS

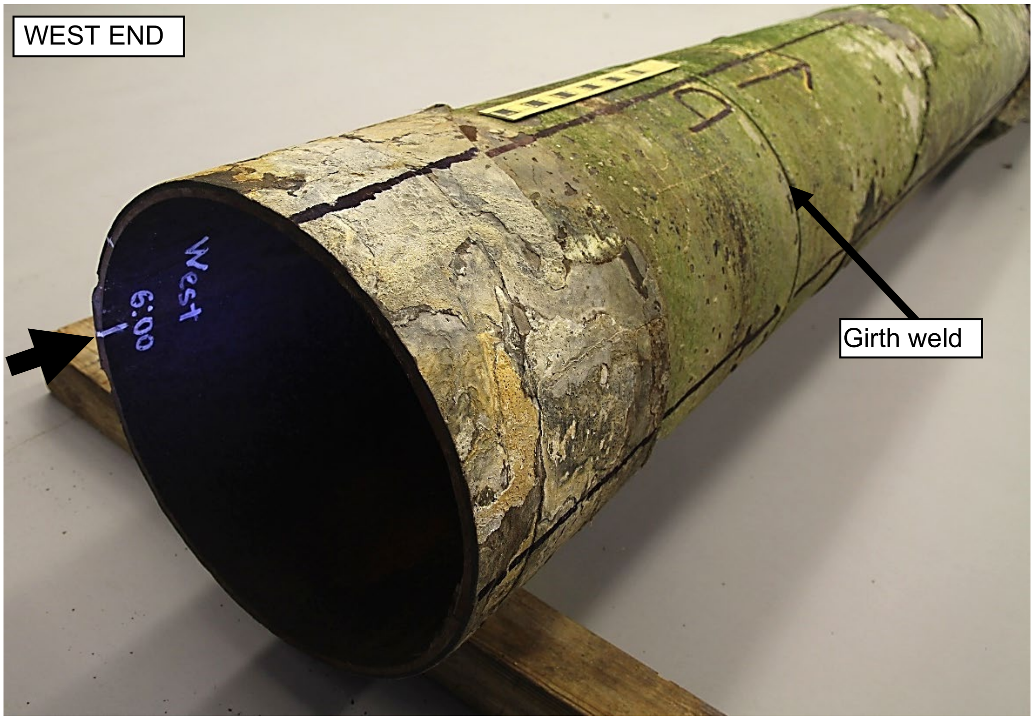
Drawing courtesy of Enterprise Products



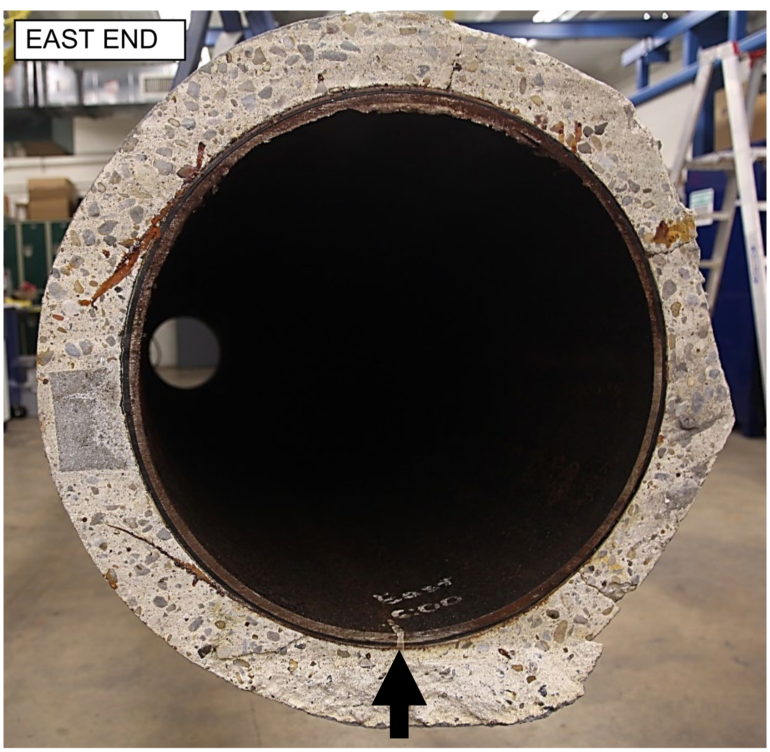
APPENDIX 2

6 O'CLOCK MARKS AT THE WEST AND EAST ENDS OF PIPE SEGMENT

(1 Page)



Appendix 2A. West cut end of the pipe showing the 6 o'clock position marked on the inner surface (indicated by an arrow).

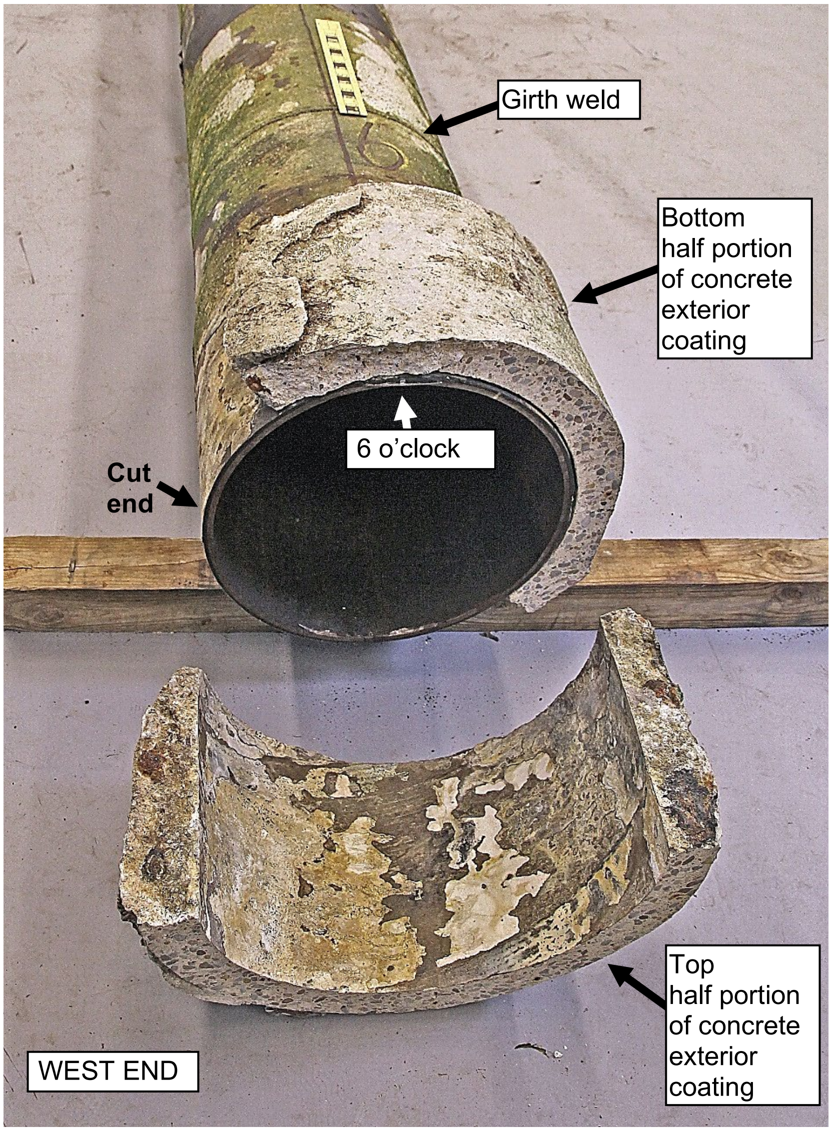


Appendix 2B. East cut end of the pipe showing the 6 o'clock position marked on the inner surface (indicated by an arrow).

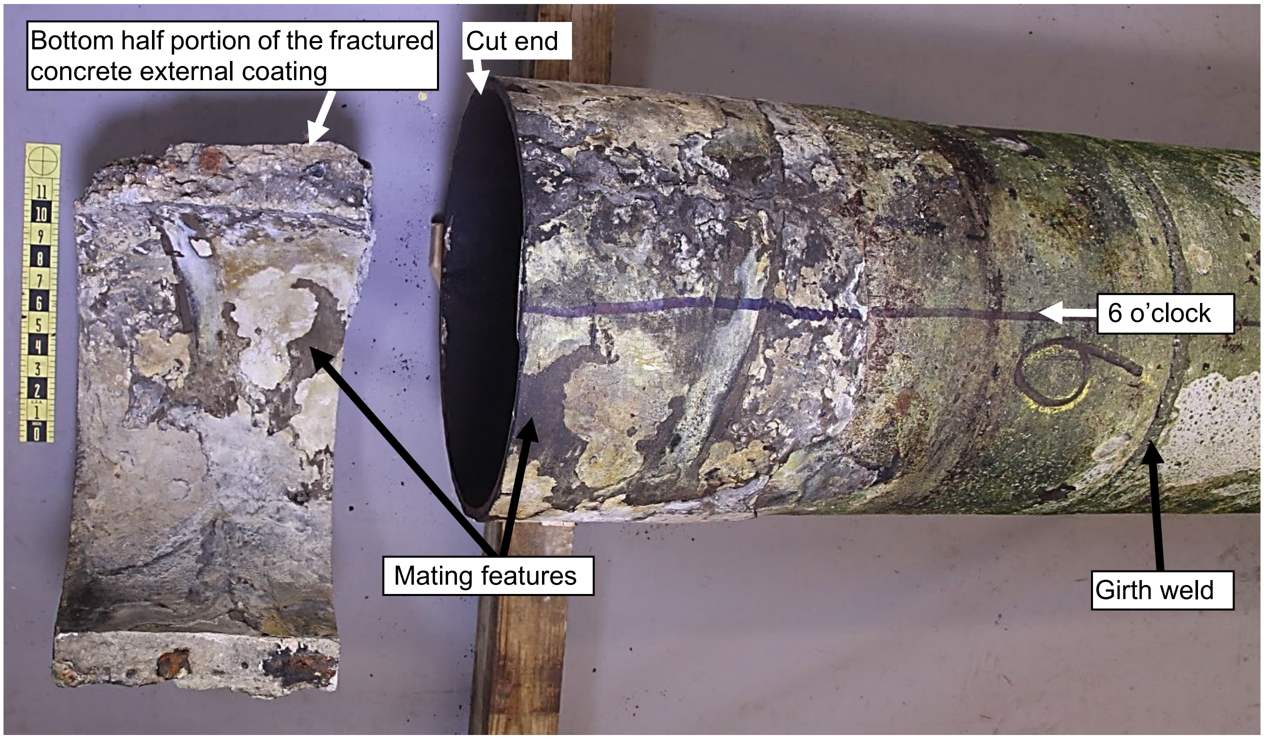
APPENDIX 3

TWO HALVES OF FRACTURED CONCREE EXTERNAL COATING
FROM WEST END OF PIPE

(Two Pages)



Appendix 3A. Photograph of the west end of the pipe segment and the two halves of a 1-foot in length concrete exterior coating that separated on-site from the west end of the pipe. The inner surface of the two concrete pieces contained marks that mated with marks found on the external surface of the pipe. This photograph shows the installed position of the bottom half portion of the broken concrete piece prior to separation. The inner surface of the top half portion of the concrete piece is shown in the foreground.

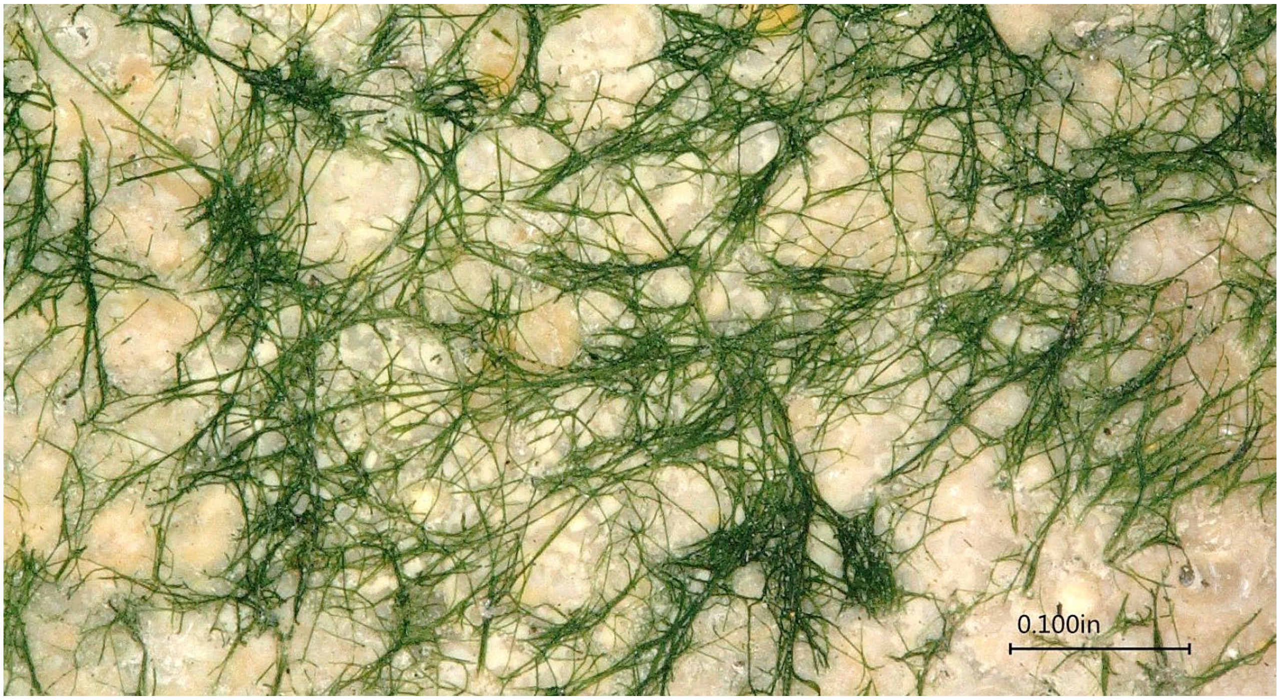


Appendix 3B. The right side of this photograph shows bottom side (6 o'clock position) of the pipe segment at the west end and the left side of this photograph shows the inner surface of the concrete exterior coating that separated from the bottom side of the pipe. Arrows show the mating features that are common between the two pieces.

APPENDIX 4

GREEN ALGEA ON OUTER SURFACE OF CALCAREOUS DEPOSIT

(1 Page)



Appendix 4. Close-up photograph of the outer surface of the calcareous deposit that appears white (background) and macro algae that appears as green branch-like features (foreground).

APPENDIX 5

X-RAY FLUORESCENCE (XRF) ANALYSIS OF CALCAREOUS DEPOSIT

(Total of 6 Pages)

EAG LABORATORIES | Eurofins Materials Science

ISO 17025

ACCREDITED

Testing Cert. #2797.01

X-RAY FLUORESCENCE (XRF)
ANALYSIS REPORT
26 Feb 2021

JOB NUMBER C0MHP460
PO NUMBER

for

Frank Zakar
National Transportation Safety Board
490 L'Enfant Plaza East
Washington, MD 20814

Prepared by:

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Reviewed by:

[redacted]

Greg Strossman, Ph.D.

[redacted]

Eurofins EAG Materials Science
810 Kifer Rd
Sunnyvale, CA 94086-5203 USA

XRF ANALYSIS REPORT

Requester:	Frank Zakar
Job Number:	C0MHP460
Analysis Date:	26 Feb 2021

Purpose:

The purpose of this analysis was to determine the composition of sample deposit flakes.

Summary:

The results are summarized in Table 1. The sample consists primarily of C, O, Mg, and Ca with trace levels of a number of other elements. These results for the major elements agree well with XRD phase identification results.

Experimental:

X-ray Fluorescence (XRF) is a non-destructive technique that can identify and quantify the elemental constituents of a sample using the secondary fluorescence signal produced by irradiation with high energy x-rays. This analysis utilized a wavelength dispersive spectrometer (WDXRF) that is capable of detecting elements from atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range up to 100% by weight.

Analytical Parameters

Instrument	Rigaku Primus II WDXRF
X-ray source	Rhodium X-ray tube
Atmosphere	Vacuum
Analysis area	20 mm diameter

Quantification was performed using the Fundamental Parameters (FP) standardless quantification software associated with the system. The fundamental parameters approach uses x-ray physics coupled with established sensitivity factors for pure elements. Relative accuracy by this method usually ranges from better than 5% up to ~20% for major elements.

Results and Interpretations:

Spectra are included in the attached figures. Sample or area names are provided on the spectra. The results are summarized in Table 1.

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https://www.eag.com/survey/?job=C0MHP460

If you would like to run further analyses on samples like those for which you have just received data, please click here: http://www.eag.com/customer-portal.html to generate a new job number and reserve your place in our queue. A customer service representative will contact you to confirm details with you soon after you fill out the short form.

For your other analytical needs please click here: http://www.eag.com/mc/contact-us-mc.html

Table 1. Sample Compositions (in Wt%)[a]

Element	Sample 1
C	12.8
O	54.8
F	0.43
Na	0.31
Mg	18.7
Al	0.15
Si	0.83
P	0.09
S	0.22
Cl	0.22
K	0.058
Ca	12.8
Ti	0.006
Mn	0.008
Fe	0.33
Ni[b]	0.001
Cu	0.001
Zn	0.003
Br	0.001
Sr	0.20

[a] The results are normalized to 100% of the measured and detected elements Note: 1.0 wt%=10,000ppm
[b] At this level, the x-ray signal may be entirely or almost entirely due to instrumental background

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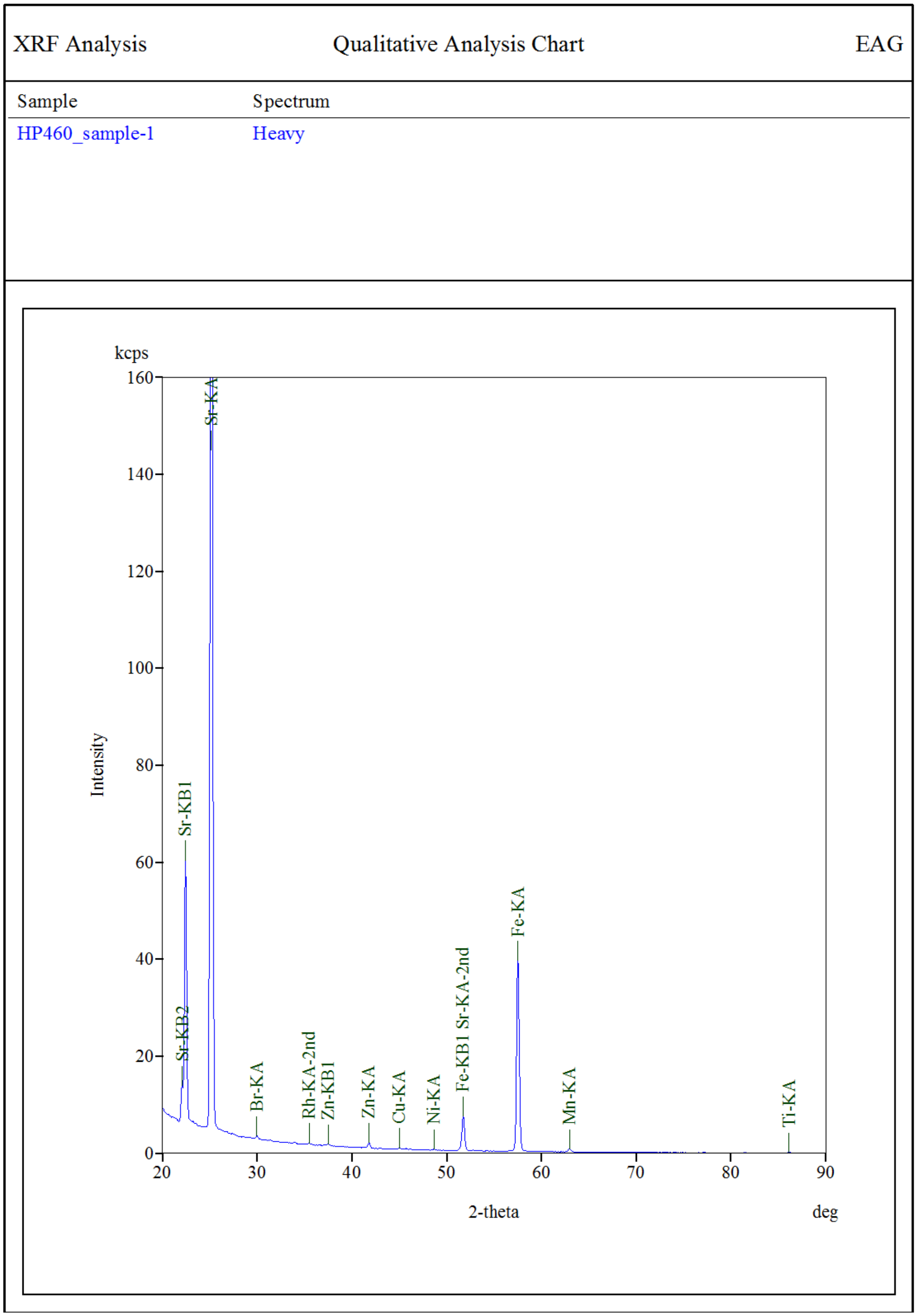
XRF Analysis
Qualitative Analysis Chart
EAG
Sample
Spectrum
HP460_sample-1
Heavy
kcps
160
140
120
100
80
60
40
20
0
Intensity
Sr-KA
Sr-KB1
Sr-KB2
Br-KA
Rh-KA-2nd
Zn-KB1
Zn-KA
Cu-KA
Ni-KA
Fe-KB1
Sr-KA-2nd
Fe-KA
Mn-KA
Ti-KA
20
30
40
50
60
70
80
90
2-theta
deg

Figure 1

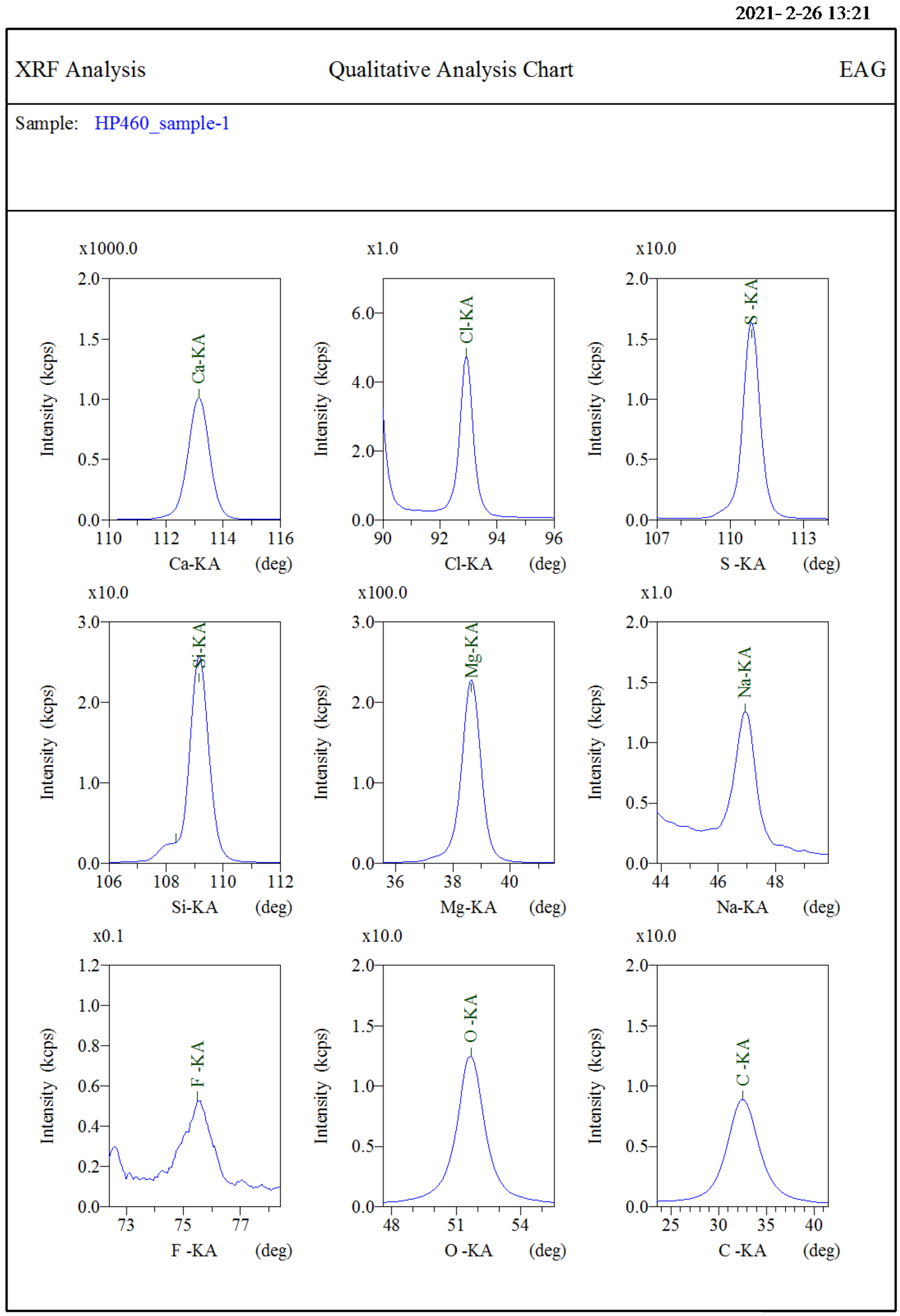
2021- 2-26 13:21
XRF Analysis
Qualitative Analysis Chart
EAG
Sample: HP460_sample-1
Intensity (kcps)
x1000.0
Ca-KA
Ca-KA (deg)
x1.0
Cl-KA
Cl-KA (deg)
x10.0
S -KA
S -KA (deg)
x10.0
Si-KA
Si-KA (deg)
x100.0
Mg-KA
Mg-KA (deg)
x1.0
Na-KA
Na-KA (deg)
x0.1
F -KA
F -KA (deg)
x10.0
O -KA
O -KA (deg)
x10.0
C -KA
C -KA (deg)

Figure 2

Wavelength Dispersive X-ray Fluorescence Spectroscopy (WDXRF) Description Appendix

In XRF photons from an x-ray tube irradiate a sample causing the ejection of inner shell electrons from the excitation volume of the sample, creating inner shell vacancies. In order to reestablish a stable electron configuration, electrons from outer shells fill the inner shell vacancies. In this process fluorescent photons are produced to balance the energy difference between the outer and inner shells. These fluorescent x-rays are the source of the signal in x-ray fluorescence spectroscopy, and their energies are characteristic of the atoms from which they originate. Therefore, the fluorescent signal can determine the elements present in the sample matrix and, from the relative intensities, the concentrations. By using an appropriate elemental and matrix reference standard, or fundamental parameter algorithms when standards are unavailable, accurate quantification of the elemental make-up of the sample can be obtained. With appropriate standards accuracies can be better than 1% relative; while using the Fundamental Parameters method typically yields accuracies of better than 5% to ~20% relative for major elements. Long term measurement reproducibility is ~2% at the 95% confidence limit.

In a wavelength dispersive XRF spectrometer (WDXRF) the fluorescence signals from the sample are collimated, after which they impinge upon one or more crystals. Each signal is diffracted at a specific angle based on the lattice spacing of the crystal and the fluorescent photon energy, following Bragg's law. Wavelength dispersive systems are generally operated by sequentially scanning the detectors over the full dispersion range of one or more crystals to collect the elemental signals. The relative intensities of the signals are a function of the concentration of the element, matrix effects, and factors attributable to the primary x-ray radiation. The system used in this analysis is capable of detecting elements of atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range to 100% by weight.

The excitation volume for XRF is both element and matrix dependent. It can range from the micrometer range for light elements in dense metallic materials to a centimeter or more for heavier elements in light element matrices such as polymers.

APPENDIX 6

X-RAY DIFFRACTION (XRD) ANALYSIS OF CALCAREOUS DEPOSIT

(Total of 6 Pages)

EAG LABORATORIES | Eurofins Materials Science

ISO 17025

ilac-MRA A2LA ACCREDITED

Testing Cert. #2797.01

X-RAY DIFFRACTION (XRD) ANALYSIS REPORT
26 Feb 2021

JOB NUMBER C0MHP460
PO NUMBER

for

Frank Zakar
National Transportation Safety Board
490 L'Enfant Plaza East
Washington, MD 20814

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Reviewed by:

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Sunnyvale, CA 94086-5203 USA

Requester:	Frank Zakar
Job Number:	C0MHP460
Analysis Date:	26 Feb 2021

X-RAY DIFFRACTION ANALYSIS REPORT

Purpose: Identify and quantify phases present in calcareous deposits removed from a submerged pipe.

Summary:

Table 1: Phase Identification and Quantification Results

Sample ID	Phases Identified	Concentration (± 5 wt%)
Calcareous deposit	$Mg(OH)_2$ – Brucite Hexagonal, SG: P-3m1 (164) PDF# [98-000-0130]	52.9 %
	$CaCO_3$ – Aragonite Orthorhombic, SG: Pmcn (62) PDF# [98-000-0098]	38.2 %
	$CaCO_3$ – Calcite Hexagonal, SG: R-3c (167) PDF# [98-000-0141]	4.0 %
	SiO_2 – Quartz Hexagonal, SG: P3221 (154) PDF# [04-016-2085]	3.9 %
	$Na_4Al_4(SiO_4)_4$ – Nepheline **??** Hexagonal, SG: P63 (173) PDF# [04-016-1737]	1.0 %

?? – Speculative identification

Experimental: The as-received samples were ground in a mortar and pestle to homogenize the samples. The resulting powders were packed into a bulk sample holder and pressed flat with a glass slide for analysis. XRD data was collected by a coupled theta: two-theta scan on a Rigaku Ultima-III diffractometer equipped with copper X-ray tube with Ni beta filter, parafocusing (Bragg-Brentano) optics, computer-controlled slits, and a 1D strip detector.

Discussion: Phase identification results are shown in Figure 1 and detailed in Table 1. This sample contains brucite, aragonite, calcite, and quartz. Nepheline was also identified, however due to the low intensity of the main observed peak and significant peak overlap elsewhere, this should be considered speculative.

Whole pattern fitting results are shown in Figure 2 and the weight percentages for each phase listed in Table 1. The R-value for this fit is 5.68%, indicating excellent agreement between the calculated fit and the observed data. The main source of error in these results is likely due to peak misfitting resulting from the large number of overlapping peaks.

Analytical Methods

Phase Identification: Crystalline phases are identified by comparing the location and relative intensity of peaks present in background modelled experimental XRD data to entries in the ICDD/ICSD diffraction database. The reference markers for the phase show where in two-theta the expected experimental peaks should be located, and the relative height of the markers indicates the expected intensity of peaks for a fine-grained and randomly oriented phase. Note that XRD is sensitive to crystal structure but relatively insensitive to elemental or chemical state composition, therefore the actual chemistry of the matching phase may differ from that of the reference card.

Semi-Quantitative Analysis: Semi-quantitative analysis is performed using WPF (whole pattern fitting), which is a subset of Rietveld Refinement that accounts for all intensity above the background curve. This technique requires that either the structure factors and atomic locations or the reference intensity ratio (a way of comparing the diffracting power of different phases) are known for all phases identified. During this process, structure factor (which relates to concentration), lattice parameters (which relate to peak position), peak width and peak shape are refined for each phase to minimize the R value – an estimate of the agreement between the model and the experimental data over the entire pattern. The WPF figures include a difference curve at the top of the pattern showing the misfit between the experimental data and modelled pattern. The difference curve is shown in a square-root intensity to accentuate the minor differences between the patterns.

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If you would like to run further analyses on samples like those for which you have just received data, please click here: http://www.eag.com/customer-portal.html to generate a new job number and reserve your place in our queue. A customer service representative will contact you to confirm details with you soon after you fill out the short form.

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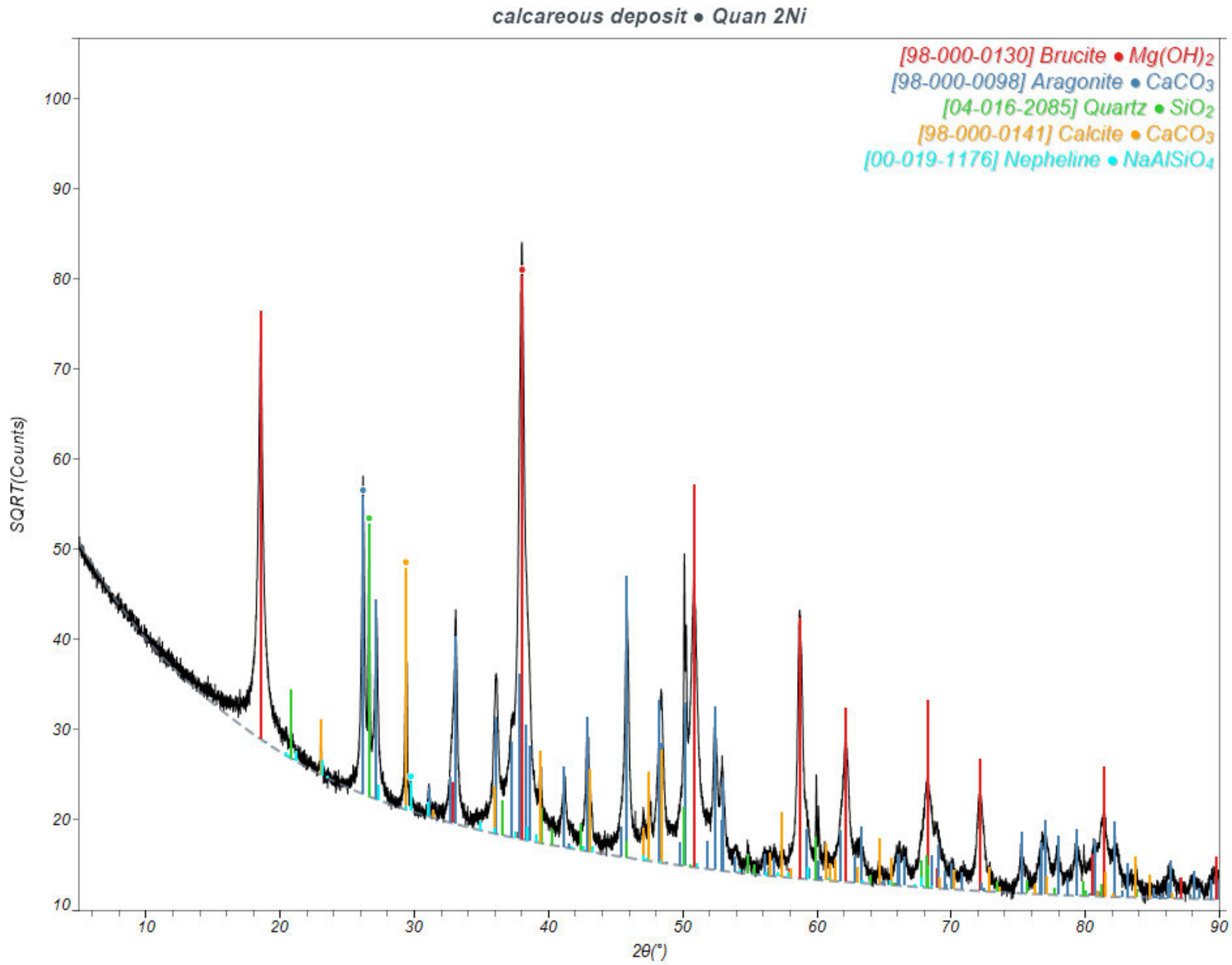
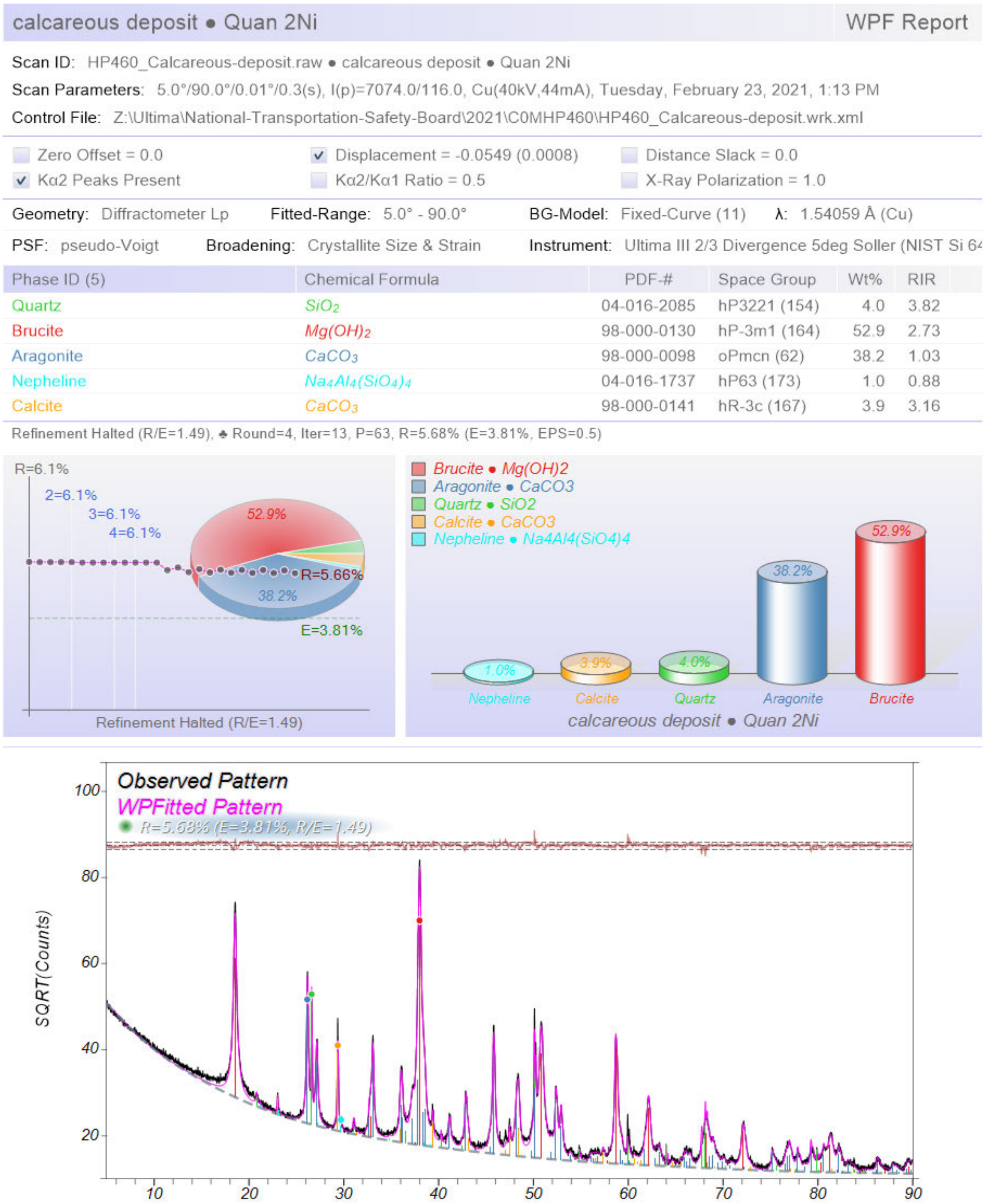


Figure 1: Phase identification for calcareous deposit



calcareous deposit • Quan 2Ni | WPF Report

Scan ID: HP460_Calcareous-deposit.raw • calcareous deposit • Quan 2Ni

Scan Parameters: 5.0°/90.0°/0.01°/0.3(s), I(p)=7074.0/116.0, Cu(40kV,44mA), Tuesday, February 23, 2021, 1:13 PM

Control File: Z:\Ultima\National-Transportation-Safety-Board\2021\C0MHP460\HP460_Calcareous-deposit.wrk.xml

Zero Offset = 0.0 | ✓ Displacement = -0.0549 (0.0008) | Distance Slack = 0.0

✓ Kα2 Peaks Present | Kα2/Kα1 Ratio = 0.5 | X-Ray Polarization = 1.0

Geometry: Diffractometer Lp | Fitted-Range: 5.0° - 90.0° | BG-Model: Fixed-Curve (11) | λ: 1.54059 Å (Cu)

PSF: pseudo-Voigt | Broadening: Crystallite Size & Strain | Instrument: Ultima III 2/3 Divergence 5deg Soller (NIST Si 64

Phase ID (5)	Chemical Formula	PDF-#	Space Group	Wt%	RIR
Quartz	SiO_2	04-016-2085	hP3221 (154)	4.0	3.82
Brucite	$Mg(OH)_2$	98-000-0130	hP-3m1 (164)	52.9	2.73
Aragonite	$CaCO_3$	98-000-0098	oPmcn (62)	38.2	1.03
Nepheline	$Na_4Al_4(SiO_4)_4$	04-016-1737	hP63 (173)	1.0	0.88
Calcite	$CaCO_3$	98-000-0141	hR-3c (167)	3.9	3.16

Refinement Halted (R/E=1.49), ♣ Round=4, Iter=13, P=63, R=5.68% (E=3.81%, EPS=0.5)

Figure 2: Whole pattern fitting results for calcareous deposit

Appendix

Measurement Uncertainty:

There are two types of uncertainty in XRD analysis; uncertainty in the number of X-ray counts at a particular angle and uncertainty in the diffraction angle. Because the arrival of X-ray quanta in the detector is random with respect to time, the accuracy of X-ray counting rate measurements is governed by the laws of probability. In particular, the size of the one sigma standard deviation in an X-ray measurement is equal to the square root of the number of X-rays counted. A conservative criterion for the detection of a weak peak in a XRD pattern must have amplitude of greater than three standard deviations above background. As a result, the more slowly a measurement is made, the lower the relative standard deviation in the number of counts measured and the more likely is detection of trace diffraction peaks. If X-ray data is acquired at a constant speed, the relative standard deviation for the major diffraction peaks in a pattern will be on the order of a few percent or less while the relative standard deviation for the weaker peaks in a pattern will be on the order of tens of percent or more. This also implies that the uncertainty in the concentrations of the major phases in a sample will be lower than for the trace phases. Please note that there are a number of sample related factors that can influence peak intensity. These include (but are not limited to): average crystallite size, preferred orientation (texture), strain, and absorption.

Uncertainty in the position of X-ray diffraction peaks is due to both instrumental and sample effects. Instrumental position uncertainty is primarily due to diffractometer misalignment. Repeat measurements of NIST standard reference materials has shown that the maximum positional uncertainty is less than +/- 0.05 degrees 2-Theta and is typically much less than that. Positional uncertainty due to sample effects are related to sample displacement (displacement of the sample surface either above or below the diffractometer focusing circle) and sample transparency (the effect gets larger as the sample matrix becomes more transparent to the incident X-rays. Through careful sample preparation, the uncertainty due to these two sample effects should be less than +/- 0.03 degrees 2-Theta. Please note that in addition to these factors, solid solution effects, where one element is partially substituted for another within a given crystal structure, can produce significant shifts in measured peak positions. Unlike sample and instrumental peak position effects, solid solution effects can result in phase misidentification.