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NACE TM 0284

EVALUATION OF PIPELINE AND PRESSURE VESSEL STEELS FOR RESISTANCE TO HYDROGEN-INDUCED CRACKING

Program: SQ-0038

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1. FOREWORD

This report summarizes the results of the **SQ-0038** proficiency testing program on the evaluation of the resistance of pressure vessel plate steels to HIC caused by hydrogen absorption from aqueous sulfide corrosion. This program is conducted in a bilateral format, following the “split-sample” design described in clause A.4.2 of ISO/IEC 17043: 2023 (Alternative interlaboratory comparisons).

South Quality conducted the testing program in April 2026 with the aim of assessing the laboratory's ability to competently perform the designated tests.

2. ORGANIZATION

Program Coordinator: Eng. Alfredo Schmidt
 Assistant Technician: Sergio Andrada
 Statistics: Lic. Manuel Tozaki
 Supervision: Eng. Emiliano Medina

3. OBJECTIVE

The objective of this proficiency testing program is the evaluation of the resistance of pressure vessel plate steels to HIC caused by hydrogen absorption from aqueous sulfide corrosion, using the following standard:

Standard
ANSI/NACE TM0284: 2016

For verification, batches of metallic plates have been selected.

Participants in this program have not been previously informed about the expected behavior of the samples they receive.

4. PARTICIPANT

Company: **METALLURGICAL RESEARCH INSTITUTE**
 Laboratory: **ANALYSIS AND TESTING LABORATORY**
 Country: Romania
 Client ID: E468
 Contact person: Marian Bratu
 General Manager
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5. HOMOGENEITY

Several batches were prepared identically by the staff at South Quality.

Subsequently, a homogeneity study was conducted to verify the CSR, CLR, and CTR parameters using an ISO/IEC 17025 accredited laboratory.

The control process followed ISO 33405: 2024, clauses 7.4.1.1 / 7.4.1.2. Stratified random sampling was applied, and samples were selected using random number generation software.

The results of this test are presented below:

Size of each batch: **100 units**

Tested samples from each batch: **20 units**

DETERMINATION	HOMOGENEITY OF RESULTS IN THE ANALYZED SAMPLES		
	BATCH: LM3122	BATCH: LM3123	BATCH: LM3124
CSR	YES	NO	YES
CLR	YES	NO	YES
CTR	YES	NO	YES

Size of each batch: **100 units**

Tested samples from each batch: **20 units**

DETERMINATION	HOMOGENEITY OF RESULTS IN THE ANALYZED SAMPLES		
	BATCH: LM3231	BATCH: LM3232	BATCH: LM3233
CSR	NO	YES	YES
CLR	YES	YES	YES
CTR	NO	YES	YES

Samples for this program are taken from selected batches identified as **LM3124** and **LM3232**.

For the batches indicated, the values determined in the homogeneity study are used as assigned values.

Analysis of this test data indicated that the selected samples were sufficiently homogeneous for the program and therefore the results of participants identified as outliers cannot be attributed to sample variability.

6. SAMPLE INFORMATION

The following samples were sent for testing:

Batch:	LM3124
Sample ID:	18 + 52 + 82
Characteristics:	Low carbon steel (SAE 1020) - 100 mm x 20 mm x 9.52 mm

Batch:	LM3232
Sample ID:	17 + 49 + 89
Characteristics:	Stainless steel (AISI 304) - 100 mm x 20 mm x 9.52 mm

7. IMAGES

SAMPLES	
	
	

8. ASSIGNED VALUES

PARAMETER	BATCH: LM3124		BATCH: LM3232	
	AVG	SD	AVG	SD
CSR	0.0259	0.0095	0	-
CLR	3.2516	0.9523	0	-
CTR	0.7923	0.2615	0	-

9. PARTICIPANT RESULTS (SEE APPENDIX)

PARAMETER	BATCH: LM3124 (AVG)	BATCH: LM3232 (AVG)
CSR	0.0360	0
CLR	3.6016	0
CTR	1.1837	0

10. STATISTICS

The results must be treated as quantitative.

According to B.4.1.3 of ISO 17043 and the appropriate technique is to compare participant results with the assigned values.

- a) When the value to compare is **zero**, the comparison is made through the difference **D** (B.1 - ISO 17043).

$$D = (x - X)$$

x is the participant's result

X is the assigned value

The performance evaluation is carried out with the following criteria:

$|D| = 0$ indicates "satisfactory" performance and generates no signal;

$|D| \neq 0$ indicates "unsatisfactory" performance and generates an action signal;

b) When the value to compare is different from **zero**, the comparison is made through **z score** (B.3- ISO 17043).

$$z = \frac{x - X}{\hat{\sigma}}$$

x is the participant's result

X is the assigned value

$\hat{\sigma}$ is the standard deviation

The performance evaluation is carried out with the following criteria:

$|z| \leq 2.0$ indicates “satisfactory” performance and generates no signal;

$2.0 < |z| < 3.0$ indicates “questionable” performance and generates a warning signal;

$|z| \geq 3.0$ indicates “unsatisfactory” performance and generates an action signal;

11. EVALUATION OF PERFORMANCE

BATCH	PARAMETER	AVERAGE		z score	PERFORMANCE RESULT
		PARTICIPANT RESULT	ASSIGNED VALUE		
LM3124	CSR	0.0360	0.0259	1.06	SATISFACTORY
	CLR	3.6016	3.2516	0.37	SATISFACTORY
	CTR	1.1837	0.7923	1.50	SATISFACTORY

BATCH	PARAMETER	AVERAGE		D	PERFORMANCE RESULT
		PARTICIPANT RESULT	ASSIGNED VALUE		
LM3232	CSR	0	0	0	SATISFACTORY
	CLR	0	0	0	SATISFACTORY
	CTR	0	0	0	SATISFACTORY

12. CONCLUSIONS

The overall performance on this **SQ-0038** program from the participant laboratory **METALLURGICAL RESEARCH INSTITUTE - ANALYSIS AND TESTING LABORATORY**, is **SUFFICIENT** based on expected results.

The criteria used for the evaluation of the overall performance are the following:

- **SUFFICIENT** performance: No unsatisfactory/questionable results were obtained.
- **ALMOST SUFFICIENT** performance: No unsatisfactory results were obtained, but one questionable result was found.
- **INSUFFICIENT** performance: An unsatisfactory result or two questionable results were obtained.

APPENDIX

PARTICIPANT RESULTS

(Results form)



INSTRUCTIONS & RESULTS FORM

PROGRAM:	Evaluation of pipeline and pressure vessel steels for resistance to hydrogen-induced cracking
CODE:	SQ-0038
VERSION:	-
STANDARD:	NACE TM 0284
COORDINATOR:	Eng. Alfredo Schmidt (aschmidt@ptsouthquality.com)

1 - General

This document serves as a guide for managing the results of the **SQ-0038** program.

Results must be typed, not handwritten.

2 - Standard

ANSI/NACE TM0284: 2016

3 - Tests involved

TEST
Evaluation of the resistance of pressure vessel plate steels to HIC caused by hydrogen absorption from aqueous sulfide corrosion

4 - Samples

CODE	SAMPLE	QUANTITY
LM3124-XX	Steel - 100 mm x 20 mm x 9.52 mm	3
LM3232-XX	Steel - 100 mm x 20 mm x 9.52 mm	3

5 - Notes

- a) Being a bilateral program, there is no deadline for submitting results.
- b) The tables in this document may be modified by the participant, if desired, to include data or observations.
- c) The samples are to be handled as routine lab samples, with all testing, documentation, and reporting adhering to **ANSI/NACE TM0284**.
- d) Samples must be retained until the end of the program, which concludes with the submission of the final report.
- e) To review the results, test images would be appreciated. Images can be attached at the end of this document or sent by email.
- f) Once this document is completed, it must be converted into a PDF file and sent to the program coordinator.

6 - Test results

Test date:	20.04.2026 – 24.04.2026	Solution:	A
Sample preparation:	Grinding <input type="checkbox"/>	Test duration:	96 h
	Sawing <input checked="" type="checkbox"/>	pH at start of test:	2.70
	Machining <input checked="" type="checkbox"/>	pH at end of test:	3.68
Polished grade:	Up to mirror brightness	Solution temperature:	25.5C deg
Method of testing of adequacy degreasing:	With acetone until proper degreasing according to ASTM F21	H ₂ S concentration (mg/L):	2369
		Purged inert gas:	Argon

SAMPLE	DIMENSIONS (mm)		
	LENGTH	WIDE	THICKNESS
LM3124-20	99.17	18.92	9.28
LM3124-57	98.17	18.94	9.24
LM3124-90	98.47	18.99	9.25

SAMPLE	SECTION	RESULTS (%)		
		CSR	CLR	CTR
LM3124-20	I	0.0204	4.4181	1.2156
	II	0.0027	1.2931	0.2114
	III	0.0219	3.4483	0.6342
LM3124-57	I	0.0234	4.4372	0.5280
	II	0.0416	2.8139	1.4784
	III	0.0427	3.6797	1.1616
LM3124-90	I	0.0000	0.0000	0.0000
	II	0.0643	4.8649	2.4223
	III	0.1070	7.4595	3.0016
MEAN		0.0360	3.6016	1.1837





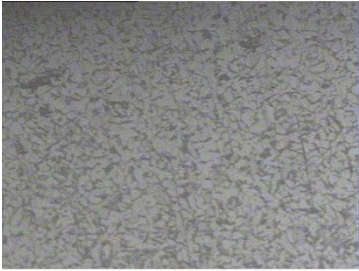

OBSERVATIONS
Alumina (Al ₂ O ₃) suspension of 0,3 µm was used to prepare by polishing the samples surfaces up to mirror brightness before optical microcope investigation. The samples tested in NACE solution A before microstructure analysis were etched with Nital solution (5 ml HNO ₃ : 95 ml etanol) for 15 seconds.

Test date:	20.04.2026 – 24.04.2026	Solution:	B
Sample preparation:	Grinding <input type="checkbox"/>	Test duration:	96 h
	Sawing <input checked="" type="checkbox"/>	pH at start of test:	8.16
	Machining <input type="checkbox"/>	pH at end of test:	5.37
Polished grade:	Up to mirror brightness	Solution temperature:	26 C deg
Method of testing of adequacy degreasing:	With acetone until proper degreasing according to ASTM F21	H ₂ S concentration (mg/L):	2318
		Purged inert gas:	Argon

SAMPLE	DIMENSIONS (mm)		
	LENGTH	WIDE	THICKNESS
LM3232-12	98.83	19.60	10.45
LM3232-51	99.18	19.46	10.01
LM3232-94	100.04	19.49	10.01

SAMPLE	SECTION	RESULTS (%)		
		CSR	CLR	CTR
LM3232-12	I	0.0000	0.0000	0.0000
	II	0.0000	0.0000	0.0000
	III	0.0000	0.0000	0.0000
LM3232-51	I	0.0000	0.0000	0.0000
	II	0.0000	0.0000	0.0000
	III	0.0000	0.0000	0.0000
LM3232-94	I	0.0000	0.0000	0.0000
	II	0.0000	0.0000	0.0000
	III	0.0000	0.0000	0.0000
MEAN		0.0000	0.0000	0.0000


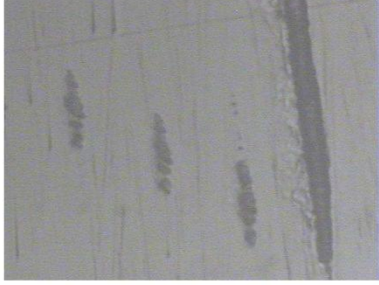

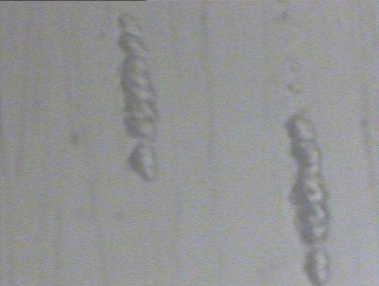




OBSERVATIONS
<p>The initial pH of the NACE B solution was 8.16. After the saturation in H₂S the pH was 5.10 and at the ending of test was 5.37. Alumina (Al₂O₃) suspension of 0,3 µm was used to prepare by polishing the samples surfaces up to mirror brightness before optical microscope investigation. The samples tested in NACE solution B before microstructure analysis were not etched. They were investigated on surfaces wiped with alcohol and dried.</p> <p>Vertical filiform corrosion in the form of putting lines is observe don the Surface of sample LM 3232 – 51. This type of filiform corrosion looks groove and is not a typical HIC crack. The active corrosion head is round and not sharp as in the case of typical HIC cracking in steps.</p> <p>On sample LM 3232- 94, to initiations of horizontal filiform corrosion are observed.</p>

PHOTOGRAPHS	
Solution A	Solution B
LM3124- 20// 57 // 90, sample surface as received	LM3232 - 12 // 51 // 64, sample surface as received
	
Surface pattern after NACE TM 0284 test	Surface pattern after NACE TM 0284 test
	
LM3124-20	LM3232-12
Microstructure of sample 20 Magnification x 100	Microstructure of sample 12 Magnification x 100
	
Microstructure of sample 20 Magnification x 200	Microstructure of sample 12 Magnification x 200
	

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LM3124- 57	LM3232-51
Microstructure of sample 57 Magnification x 100	Microstructure of sample 51 Magnification x 100
	
Microstructure of sample 57 Magnification x 200	Microstructure of sample 51 Magnification x 200
	
LM3124-90	LM3232-94
Microstructure of sample 90 Magnification x 100	Microstructure of sample 94 Magnification x 50
	
Microstructure of sample 90 Magnification x 200	Microstructure of sample 94 Magnification x 100
	

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