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NACE TM 0284

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

Program: SQO-M4 Round 17

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1. FOREWORD

This report summarizes the results of the **SQO-M4 (Round 17)** 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 carried out under a simultaneous participation format, according to the A.3.1 classification of the ISO 17043 standard ("Model 2 - Figure A.1").

South Quality conducted the testing program between June and August 2025. The aim of the program was to assess laboratory ability to competently perform the nominated tests.

2. ORGANIZATION

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

3. OBJECTIVE

The objective of this proficiency testing program is to evaluate 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

Batches of steels samples have been selected.

Participants in this program have not been previously informed of the values or the expected range of values for the samples they will receive.

4. PARTICIPANTS

In the present round, 22 laboratories have participated with the following details:

CODE	Country	ISO 17025 Accredited	Results delivered
01	Brazil	Yes	Yes
02	Malaysia	Yes	Yes
03	France	Yes	No
04	Spain	No	Yes
05	Peru	No	Yes
06	Brazil	Yes	Yes
07	Spain	Yes	No
08	Portugal	Yes	Yes
09	Spain	Yes	Yes
10	Pakistan	No	Yes
11	Italy	Yes	Yes
12	Mexico	Yes	Yes
13	Türkiye	Yes	Yes
14	Argentina	Yes	No
15	Colombia	Yes	Yes
16	Australia	Yes	Yes
17	Germany	Yes	Yes
18	England	Yes	Yes
19	Romania	Yes	Yes
20	Hong Kong	Yes	No
21	Germany	Yes	Yes
22	France	No	Yes

5. HOMOGENEITY

Several batches were prepared identically by South Quality staff.

Subsequently, a homogeneity study was conducted with an ISO 17025 accredited laboratory.

The control process followed ISO Guide 35: 2017, clause 7.4.1.2. Stratified random sampling was employed, and samples were chosen using random number generation software.

The results of these tests are presented below:

Size of each batch: **150 samples** Tested samples from each batch: **30 samples**

DETERMINATION (SOLUTION A)	HOMOGENEITY OF RESULTS IN THE ANALYZED SAMPLES - LOW CARBON STEEL -		
	BATCH: LM3419	BATCH: LM3420	BATCH: LM3421
CSR	YES	YES	YES
CLR	YES	YES	YES
CTR	YES	YES	YES

Size of each batch: **150 samples** Tested samples from each batch: **30 samples**

DETERMINATION (SOLUTION B)	HOMOGENEITY OF RESULTS IN THE ANALYZED SAMPLES - STAINLESS STEEL -		
	BATCH: LM3442	BATCH: LM3528	BATCH: LM3529
CSR	NO	NO	YES
CLR	NO	NO	YES
CTR	NO	NO	YES

Samples for this program are taken from selected batches identified as **LM3421** and **LM3528**.

Analysis of this testing data indicated that samples were sufficiently homogeneous for the program and, therefore, any participant results identified as outliers cannot be attributed to sample variability.

6. SAMPLE INFORMATION

The following samples were sent for testing (Participant **Code 17**):

Batch:	LM3421
Sample ID:	23 + 76 + 135
Characteristics:	Carbon steel (SAE 1010) - 100 x 19.1 x 9.5 mm

Batch:	LM3528
Sample ID:	09 + 72 + 140
Characteristics:	Stainless steel (AISI 304) - 100 x 19.1 x 9.5 mm

7. IMAGES

SAMPLES

8. ASSIGNED VALUES

The assigned values are obtained from the results reported by all participants (**Consensus values**).

9. PARTICIPANTS' RESULTS

LABORATORY CODE	LM3421			LM3528		
	MEAN			MEAN		
	CSR	CLR	CTR	CSR	CLR	CTR
01	0.250	2.591	5.448	0	0	0
02	0.117	4.247	3.516	0	0	0
04	1.131	4.112	3.275	0	0	0
05	1.389	2.335	9.523	0	0	0
06	0.336	4.001	5.207	0	0	0
08	1.445	2.014	3.161	0	0	0
09	0.659	2.591	2.866	0	0	0
10	0.781	5.953	2.636	0	0	0
11	0.488	2.719	2.225	0	0	0
12	0.913	6.859	3.123	0	0.05	0.04
13	1.051	2.847	2.495	0	0	0
15	1.378	4.045	3.874	0	0	0
16	1.168	1.472	2.886	0	0	0
17	1.680	4.600	6.220	0	0.03	0.09
18	1.304	1.640	5.442	0	0	0
19	0.398	0.654	0.007	0	0	0
21	0.420	4.470	6.950	0	0	0
22	1.472	1.632	4.915	0	0	0

ASSIGNED VALUES				
PARAMETER	LM3421		LM3528	
	AVG	SD	AVG	SD
CSR	0.910	0.490	0	-
CLR	3.054	1.407	0	-
CTR	4.098	2.148	0	-

10. STATISTICS

The results must be treated as qualitative as quantitative.

According B.3.1.3 of ISO 17043 the appropriate technique is to compare participant results with the assigned values.

a) For qualitative results (Values equal to zero), the comparison will be made directly against the assigned value, and any difference will be evaluated as **Unsatisfactory**.

b) For quantitative results the comparison will be made through ***z score*** (B3 - 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 of each sample 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

LABORATORY CODE	| *z score* | - BATCH: LM3421		
	CSR	CLR	CTR
01	1.35	0.33	0.63
02	1.62	0.85	0.27
04	0.45	0.75	0.38
05	0.98	0.51	**2.53**
06	1.17	0.67	0.52
08	1.09	0.74	0.44
09	0.51	0.33	0.57
10	0.26	**2.06**	0.68
11	0.86	0.24	0.87
12	0.20	**3.36**	0.26
13	0.29	0.15	0.75
15	0.96	0.70	0.10
16	0.53	1.12	0.56
17	1.88	1.55	1.41
18	0.80	1.00	0.63
19	0.92	1.59	1.95
21	0.88	1.45	1.80
22	1.15	1.01	0.38

Laboratory **Code 01**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 02**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 03**: The laboratory has not sent the results before the deadline.

Laboratory **Code 04**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 05**: The laboratory obtained **QUESTIONABLE** results for the CTR parameter determination in the sample from batch LM3421. However, **SATISFACTORY** results were achieved for all other parameters in both samples.

Laboratory **Code 06**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 07**: The laboratory has not sent the results before the deadline.

Laboratory **Code 08**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 09**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 10**: The laboratory obtained **QUESTIONABLE** results for the CLR parameter determination in the sample from batch LM3421. However, **SATISFACTORY** results were achieved for all other parameters in both samples.

Laboratory **Code 11**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 12**: The laboratory obtained **UNSATISFACTORY** results for the CLR parameter determination in the sample from batch LM3421 and for the CLR and CTR parameters in the sample from batch LM3528. However, **SATISFACTORY** results were achieved for all other parameters in both samples.

Laboratory **Code 13**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 14**: The laboratory has not sent the results before the deadline.

Laboratory **Code 15**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 16**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 17**: The laboratory obtained **UNSATISFACTORY** results for the CLR and CTR parameters in the sample from batch LM3528. However, **SATISFACTORY** results were achieved for all other parameters in both samples.

Laboratory **Code 18**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 19**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 20**: The laboratory has not sent the results before the deadline.

Laboratory **Code 21**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

Laboratory **Code 22**: The laboratory obtained **SATISFACTORY** results for the determination of all assigned values in both samples.

12. CONCLUSIONS

The overall performance of participating laboratories in the **SQO-M4 (Round 17)** program, based on expected results, is summarized as follows:

- Laboratories Codes **01, 02, 04, 06, 08, 09, 11, 13, 15, 16, 18, 19, 21,** and **22** have obtained a **SUFFICIENT** performance according to the expected results and should not take any action;

- Laboratories Codes **05,** and **10** has obtained an **ALMOST SUFFICIENT** performance according to the expected results and must evaluate whether it is necessary to take action on the tests where they obtained a different result than expected;

- Laboratory Code **12**, and **17** has obtained an **INSUFFICIENT** performance according to the expected results and must take action on the tests where they obtained a different result than expected (See Appendix B).

The criteria used for evaluating overall performance are as follows:

- **SUFFICIENT** performance: No unsatisfactory or questionable results were obtained.

- **ALMOST SUFFICIENT** performance: No unsatisfactory results were obtained, but one questionable result was found.

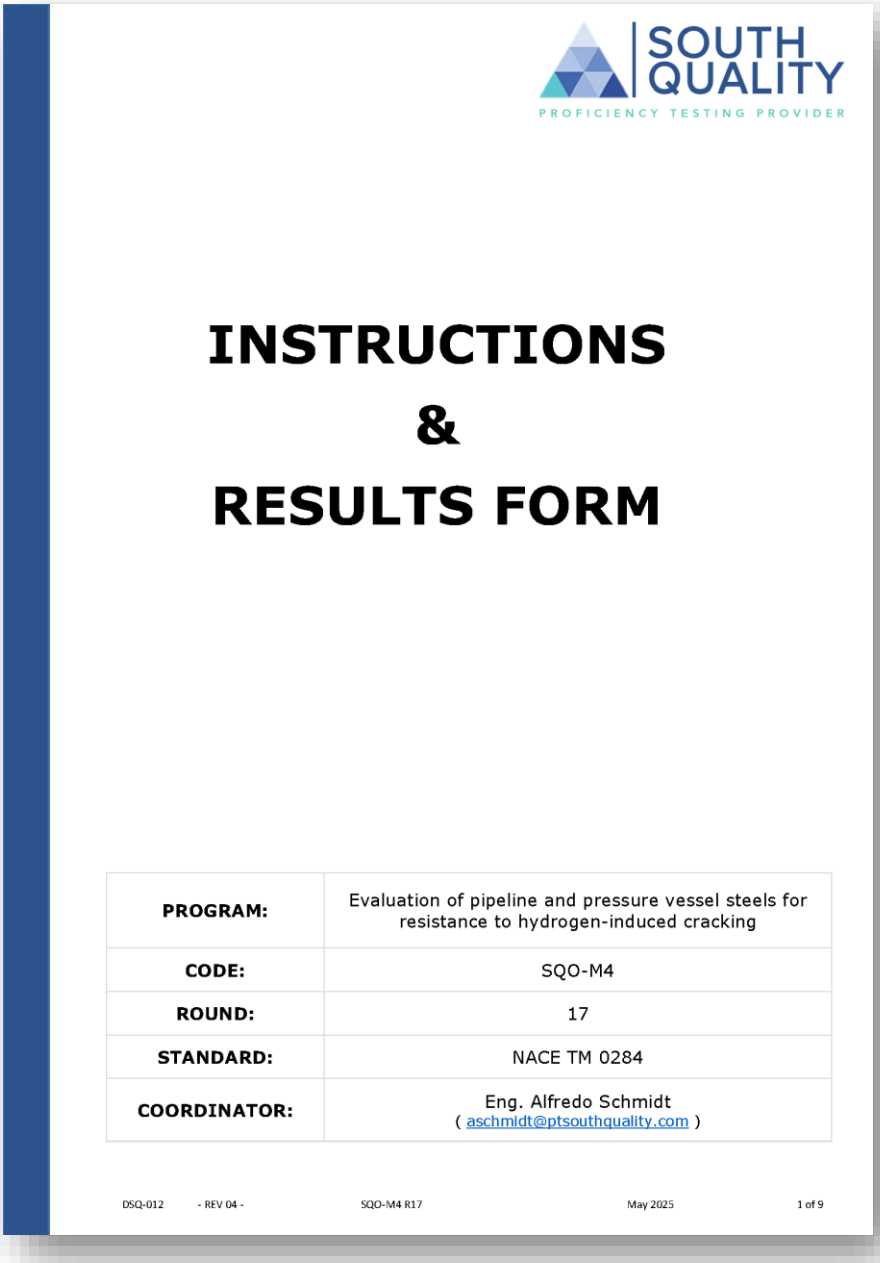
- **INSUFFICIENT** performance: One unsatisfactory result or two questionable results were obtained.

APPENDIX A

A1 - PARTICIPANT DATA

Company:	**SALZGITTER MANNESMANN FORSCHUNG GMBH**
City-Country:	Salzgitter - Germany
Client ID:	E518
Contact person:	Dr.-Ing. Markus Krieger
	Head of Department Materials Characterization
	m.krieger@sz.szmf.de

A2 - PARTICIPANT RESULTS



SOUTH QUALITY
PROFICIENCY TESTING PROVIDER

INSTRUCTIONS
&
RESULTS FORM

PROGRAM:	Evaluation of pipeline and pressure vessel steels for resistance to hydrogen-induced cracking
CODE:	SQO-M4
ROUND:	17
STANDARD:	NACE TM 0284
COORDINATOR:	Eng. Alfredo Schmidt (aschmidt@ptsouthquality.com)

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1 - General

This document is intended to be filled with the results of the **SQO-M4** program (Round 17).

Results must be typed, not handwritten.

2 - Standard

ANSI/NACE TM0284: 2016

3 - Participant

SALZGITTER MANNESMANN FORSCHUNG GMBH (Salzgitter)	**CODE 17**

4 - Tests involved

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

5 - Samples

CODE	SAMPLE	QUANTITY
LM3421-XX	Steel - 100 x 19.1 x 9.5 mm	3
LM3528-XX	Steel - 100 x 19.1 x 9.5 mm	3

6 - Notes

a) The deadline for the delivery of results is **July 21, 2025**.
b) Tables in this document may be modified to include additional data or observations.
c) Samples must be retained until the end of the program, which concludes with the submission of the final report.
d) Participants may improve the surface to ensure a better testing area. If so, the procedure must be described in the observations box.
e) Samples **LM3421** are to be tested using **solution A**.
f) Samples **LM3528** are to be tested using **solution B**.
g) The sample should be treated as a routine laboratory specimen. All testing, recording, and reporting must be carried out in accordance with ANSI/NACE TM0284.
h) To support the review of results, submission of test images is appreciated. Images may be attached at the end of this document or sent via email.
i) Once the document is completed, please convert it to a PDF file and send it to the program coordinator.

7 - **Test results**

Test date:	2025-06-23 (start) 2025-07-18 (end)	Solution:	**A**
Sample preparation:	Grinding ☒	Test duration:	96 h
	Sawing ☒	pH at start of test:	2,76
	Machining ☐	pH at end of test:	3,53
Polished grade:	1 µm	Solution temperature:	25,0 °C
Method of testing of adequacy degreasing:	Cleaning for 40 s with a mixture of (4 g/L Gardobond H7390 + 5 g/L NaOH) at 80 °C, followed by rinsing with distilled water and ethanol. Subsequent handling with gloves only.	H_2S concentration (mg/L):	2905 mg/L
		Purged inert gas:	N_2 (for 1 hour)

SAMPLE	DIMENSIONS (mm)		
	LENGTH	WIDE	THICKNESS
LM3421-23	≥100 *)	19,38	8,40
LM3421-76	≥100 *)	19,02	8,31
LM3421-135	≥100 *)	19,09	8,30

SAMPLE	SECTION	RESULTS (%)		
		CSR	CLR	CTR
LM3421-23	I	0,00	0,00	0,00
	II	0,00	0,52	0,24
	III	0,01	0,41	1,31
LM3421-76	I	0,14	4,31	3,25
	II	14,98	36,12	51,14
	III	0,00	0,00	0,00
LM3421-135	I	0,00	0,00	0,00
	II	0,00	0,00	0,00
	III	0,00	0,00	0,00
MEAN		**1,68**	**4,60**	**6,22**

OBSERVATIONS
Testing according to chapter 3 and 8 of NACE TM 0284:2016 was carried out by our sister company (Salzgitter Flachstahl GmbH – Chemical Laboratories: accreditation number D-PL-18292-01-01). Evaluation of samples according to chapter 8 of NACE TM 0284:2016 was carried out by our own laboratory (Salzgitter Mannesmann Forschung GmbH – Laboratory for Metallography and Physical Metallurgy; accreditation number D-PL-11278-02-00). On the outer samples surfaces some cracks were observed, which were filled by scale and partly in combination with internal oxides. These were not taken into the evaluation, since these cracks obviously result from the production process and are not initiated through the HIC testing. *) In our internal test documents, we do not report exact values of initial sample length. When the samples are delivered to the lab, we just check, if they have a minimum length of 100 mm, to ensure the minimum section length of 25 mm when cutting the sections from the sample bar.

Test date:	2025-06-23 (start) 2025-07-18 (end)	Solution:	**B**
Sample preparation:	Grinding ☒	Test duration:	96 h
	Sawing ☒	pH at start of test:	5,21
	Machining ☐	pH at end of test:	5,23
Polished grade:	1 µm	Solution temperature:	25,0 °C
Method of testing of adequacy degrasing:	Cleaning for 40 s with a mixture of (4 g/L Gardobond H7390 + 5 g/L NaOH) at 80 °C, followed by rinsing with distilled water and ethanol. Subsequent handling with gloves only.	H_2S concentration (mg/L):	3059 mg/L
		Purged inert gas:	N_2 (for 1 hour)

SAMPLE	DIMENSIONS (mm)		
	LENGTH	WIDE	THICKNESS
LM3528-09	≥100 *)	18,99	9,17
LM3528-72	≥100 *)	18,93	9,09
LM3528-140	≥100 *)	18,87	8,93

SAMPLE	SECTION	RESULTS (%)		
		CSR	CLR	CTR
LM3528-09	I	0,00	0,00	0,00
	II	0,00	0,00	0,00
	III	0,00	0,00	0,00
LM3528-72	I	0,00	0,26	0,77
	II	0,00	0,00	0,00
	III	0,00	0,00	0,00
LM3528-140	I	0,00	0,00	0,00
	II	0,00	0,00	0,00
	III	0,00	0,00	0,00
MEAN		**0,00**	**0,03**	**0,09**

OBSERVATIONS
Testing according to chapter 3 and 8 of NACE TM 0284:2016 was carried out by our sister company (Salzgitter Flachstahl GmbH – Chemical Laboratories: accreditation number D-PL-18292-01-01). Evaluation of samples according to chapter 8 of NACE TM 0284:2016 was carried out by our own laboratory (Salzgitter Mannesmann Forschung GmbH – Laboratory for Metallography and Physical Metallurgy; accreditation number D-PL-11278-02-00). *) In our internal test documents, we do not report exact values of initial sample length. When the samples are delivered to the lab, we just check, if they have a minimum length of 100 mm, to ensure the minimum section length of 25 mm when cutting the sections from the sample bar.

PHOTOGRAPHS

Bild 6 (B_876135)
00023_C
LM3421-23

100:1
poliert
Längsschliff

Example of crack in sample LM3421-23, section II (C)

Bild 8 (B_877199)
00076_B
LM3421-76

100:1
poliert
Längsschliff

Example of crack in sample LM3421-76, section I (B)

Bild 9 (B_877245)
00076_C
LM3421-76

12.5:1
poliert
Längsschliff

Example of crack in sample LM3421-76, section II (C)

APPENDIX B

VOID

----- END OF REPORT -----