SOUTH QUALITY

PROFICIENCY TESTING PROVIDER

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Status: FINAL REPORT

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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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Prepared by:	Reviewed by:	Approved by:
cn=SERGIO E. ANDRADA, o=PT SOUTH QUALITY SAS, ou=PT SOUTH QUALITY SAS, email=tech@ptsouthquality.com, c=AR 2025.08.15 16:43:20 -03'00'	cn=ALFREDO M. SCHMIDT, o=PT SOUTH QUALITY SAS, ou=SOUTH QUALITY, email=aschmidt@ptsouthquality.com, c=AR 2025.08.18 15:20:50 -03'00'	cn=Emiliano Medina, o=SOUTH QUALITY, ou=PT SOUTH QUALITY SAS, email=quality@ptsouthquality.com, c=ES 2025.08.19 11:52:07 -03'00'
Sergio Andrada Assistant Technician	**Eng. Alfredo Schmidt** Metallurgical expert	**Eng. Emiliano Medina** Quality Assurance Lead

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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

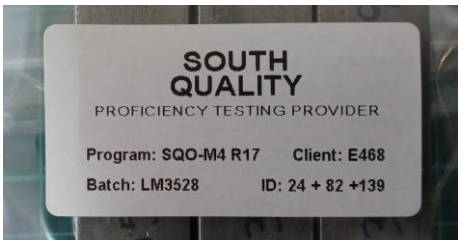
The following samples were sent for testing (Participant **Code 19**):

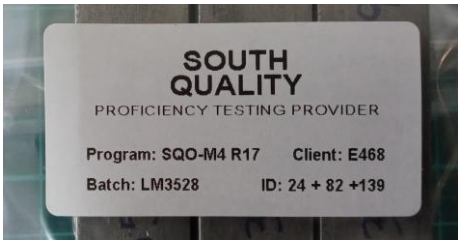
Batch:	LM3421
Sample ID:	32 + 80 + 147
Characteristics:	Carbon steel (SAE 1010) - 100 x 19.1 x 9.5 mm

Batch:	LM3528
Sample ID:	24 + 82 + 139
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.

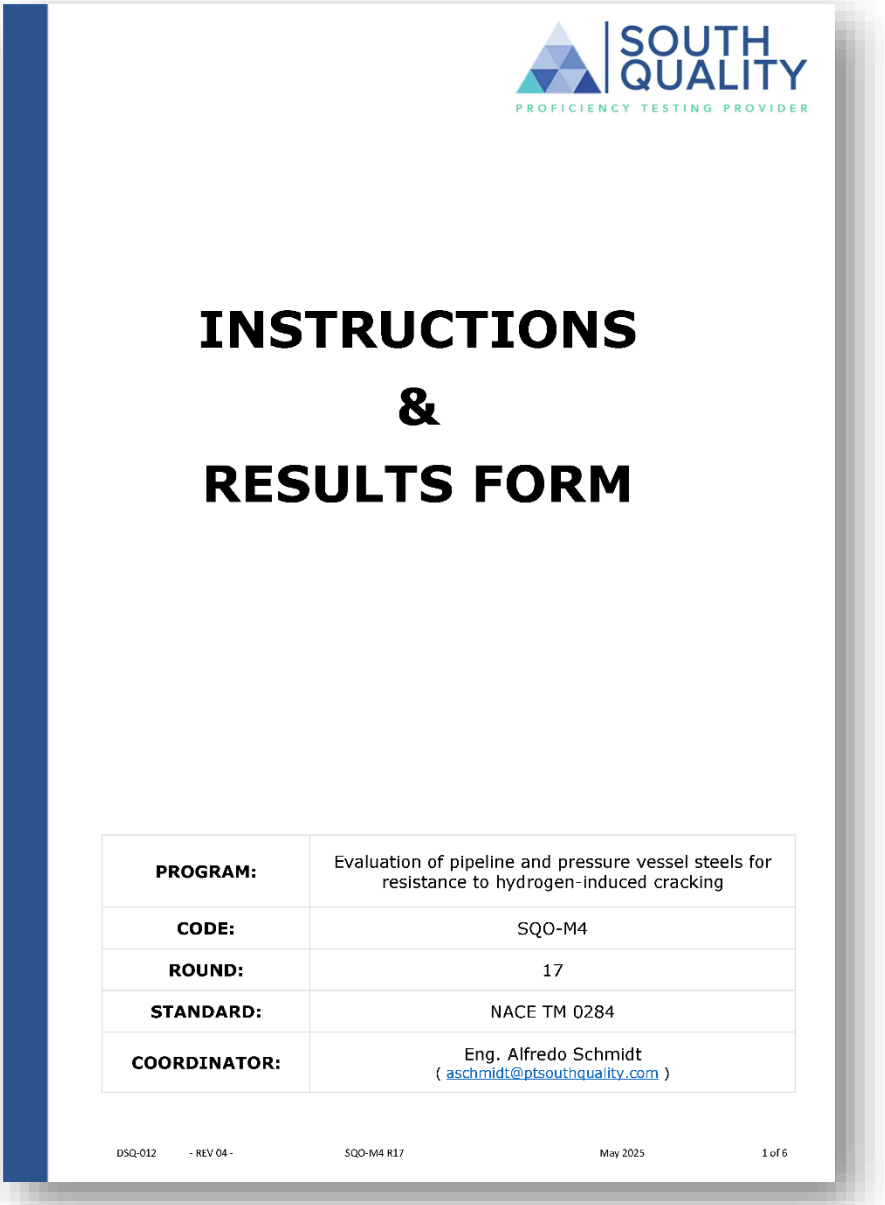
- **INSUFFICIENT** performance: One unsatisfactory result or two questionable results were obtained.

APPENDIX A

A1 - PARTICIPANT DATA

Company:	**METALLURGICAL RESEARCH INSTITUTE**
Laboratory:	**ANALYSIS AND TESTING LABORATORY**
Country:	Romania
Client ID:	E468
Contact person:	Marian Bratu - General Manager
	icem.bucuresti@yahoo.com

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**

METALLURGICAL RESEARCH INSTITUTE ANALYSIS AND TESTING LABORATORY	**CODE 19**

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:	30.06.2025 – 04.07.2025	Solution:	A
Sample preparation:	Grinding ☐	Test duration:	96 h
	Sawing ☒	pH at start of test:	2,62
	Machining ☐	pH at end of test:	3,75
Polished grade:	Up to mirror brightness	Solution temperature:	27,5 C deg
Method of testing of adequacy degreasing:	With acetone until proper degrasing according to ASTM F21	H_2S concentration (mg/L):	2505
		Purged inert gas:	Argon

SAMPLE	DIMENSIONS (mm)		
	LENGTH	WIDE	THICKNESS
LM3421-32	107,42	19,01	9,17
LM3421-80	105,98	19,06	9,26
LM3421-147	105,25	19,13	9,21

SAMPLE	SECTION	RESULTS (%)		
		CSR	CLR	CTR
LM3421-32	I	*0.4734*	*0.8724*	*0.0041*
	II	*1.6833*	*2.3991*	*0.0404*
	III	*1.4203*	*2.6172*	*0.0189*
LM3421-80	I	*0.0000*	*0.0000*	*0.0000*
	II	*0.0000*	*0.0000*	*0.0000*
	III	*0.0000*	*0.0000*	*0.0000*
LM3421-147	I	*0.0000*	*0.0000*	*0.0000*
	II	*0.0000*	*0.0000*	*0.0000*
	III	*0.0000*	*0.0000*	*0.0000*
MEAN		*0.3975*	*0.6543*	*0.0070*

OBSERVATIONS
Alumina (Al2O3) 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 HNO3 : 95 ml etanol) for 15 seconds.

Test date:	30.06.2025 – 04.07.2025	Solution:	B
Sample preparation:	Grinding ☐	Test duration:	96 h
	Sawing ☒	pH at start of test:	8,17
	Machining ☐	pH at end of test:	5,23
Polished grade:	Up to mirror brightness	Solution temperature:	27 C deg
Method of testing of adequacy degrasing:	With acetone until proper degrasing according to ASTM F21	H_2S concentration (mg/L):	2403
		Purged inert gas:	Argon

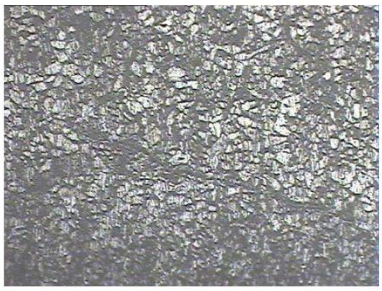
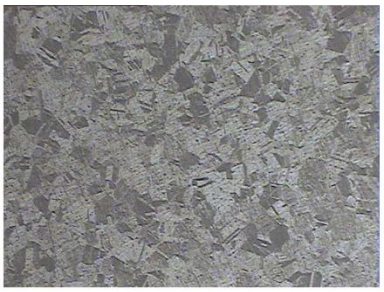
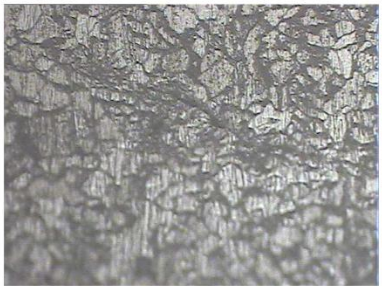
SAMPLE	DIMENSIONS (mm)		
	LENGTH	WIDE	THICKNESS
LM3528-24	100,91	18,84	9,55
LM3528-82	100,08	18,80	9,56
LM3528-139	99,57	18,78	9,56

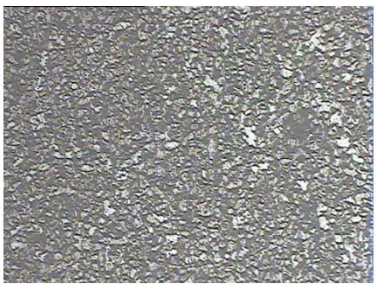
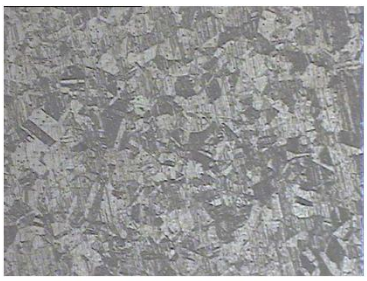
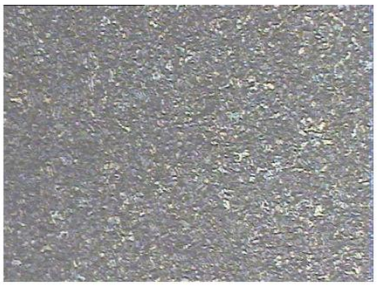
SAMPLE	SECTION	RESULTS (%)		
		CSR	CLR	CTR
LM3528-24	I	*0.0000*	*0.0000*	*0.0000*
	II	*0.0000*	*0.0000*	*0.0000*
	III	*0.0000*	*0.0000*	*0.0000*
LM3528-82	I	*0.0000*	*0.0000*	*0.0000*
	II	*0.0000*	*0.0000*	*0.0000*
	III	*0.0000*	*0.0000*	*0.0000*
LM3528-139	I	*0.0000*	*0.0000*	*0.0000*
	II	*0.0000*	*0.0000*	*0.0000*
	III	*0.0000*	*0.0000*	*0.0000*
MEAN		***0.0000***	***0.0000***	***0.0000***

OBSERVATIONS

The initial pH of the NACE B solution was 8.17. After the saturation in H2S the pH was 5.12 and at the ending of test was 5.23.
Alumina (Al2O3) 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 B before microstructure analysis were etched with aqua regia, namely solution of HCl :HNO3 : (3:1) diluted in 1:1 with distiled water during 5 minute at the room temperature.

PHOTOGRAPHS

Solution A	**Solution B**
LM3421- 32// 80 // 147	LM3428 - 24 // 82 // 139
Surface pattern after NACE TM 0284 test	Surface pattern after NACE TM 0284 test
	
LM3421-32	LM3428-24
Microstructure of sample 32 Magnification x 100	Microstructure of sample 24 Magnification x 50
	
Microstructure of sample 32 Magnification x 200	Microstructure of sample 24 Magnification x 100
	

LM3421- 80	LM3428-82
Microstructure of sample 80 Magnification x 50	Microstructure of sample 82 Magnification x 50
	
Microstructure of sample 80 Magnification x 100	Microstructure of sample 82 Magnification x 100
	
LM3421-147	LM3428-139
Microstructure of sample 147 Magnification x 50	Microstructure of sample 139 Magnification x 50
	
Microstructure of sample 147 Magnification x 100	Microstructure of sample 139 Magnification x 100
	

APPENDIX B

VOID

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