



TEST REPORT

Test Report No: 160293

Client: Gulmohar Pack-Tech India Pvt. Ltd.
Head Office & Factory
Pune- 411015 Maharashtra
INDIA

Contract No / Date: -- / 18.06.2016 and 10.08.2016

Offer No: 160279, 160291

Subcontractors: not applicable

Archiving of Samples: three months

Subject of Testing: VCI Packaging Material

Aim of Testing: Testing and Releasing of VCI Packaging Material

Origin of Samples: sent by customer via mail

Entry Date of Samples: 23.08.2016

Start of Testing: 23.08.2016

End of Testing: 01.11.2016

Laboratory: Labor Materialprüfung

Test Methods: VW 50164, Issue 2013-06^{*)}

No of Pages: 12

The test methods marked ^{*)} are non-accredited test methods



1 Subject of Testing

For examination the following sample was received, as displayed in Table 1:

Table 1. Samples

Sample Name iLF	Sample Name Customer
E-160293-P1	VCI film
E-160293-P2	LDPE film

2 Testing

2.1 Association of a reference film with the VCI film, Carrying out the following tests

1. Determination of the layer thicknesses, DIN EN ISO 2178
2. Recording of FTIR transmittance spectra, DIN EN 1767
3. Determination of the ash in mass % acc. DIN EN ISO 3451-1 *)
4. Recording of FTIR transmittance spectra of the ash
5. Recording of DSC curves, ISO 11357 part 1 to 6 *)

2.2 Analysis for the presence of secondary amines and nitrite as per Technical Rule for Hazardous Substances TRGS 615

Requirement for maximum concentration according to VW 50164, point 4.1 for the following substances:

Diethanolamine	: must not exceed a mass fraction of 0.02%
Morpholine	: must not exceed a mass fraction of 0.02%
4,4'-Methylenedimorpholine:	must not exceed a mass fraction of 0.02%
Piperazine	: must not exceed a mass fraction of 0.02%
Nitrite	: must not exceed a mass fraction of 0.1% in the finished product, a nitrite mass fraction of 0.1% to 1% is permissible only if the manufacturer declares that the VCI film cannot form or release any N-nitrosamines when used properly.

2.3 Examination of the corrosion protection effect

2.3.1 Distance test, flask test (K test) acc. VW 50164, subsection 5.1 *)

Preparation: - samples were taken from representative areas of the materials to be sampled
 - cuts with the dimensions 25 mm × (150 ± 0,5) mm were prepared; 8 cuts of VCI material and 4 cuts of the corresponding NON VCI material

Completion: - minimum 4-fold determination with VCI material; 2-fold determination with VCI-free material

Metallic test specimen:

- in humid air corrodible steel S235JRG2 (Material 1.0038, called constructional steel)

Test set: - Erlenmeyer flask and rubber plug with metallic specimens and 2 "sample Cuts" as a closure for Erlenmeyer flask (temporarily filled with glycerol-water mixture)

Test cycle: - (20 ± 0.5) hours at (23 ± 2C) Erlenmeyer flask empty
 - Filling Flask with glycerol-water mixture
 - (2h ± 10min) at (23 ± 2) ° C
 - (2h ± 10min) at (40 ± 1) ° C

Evaluation: - Analyses of the surface state of the steel specimen according diagrams VW 50164 picture 3 and the following figures 1

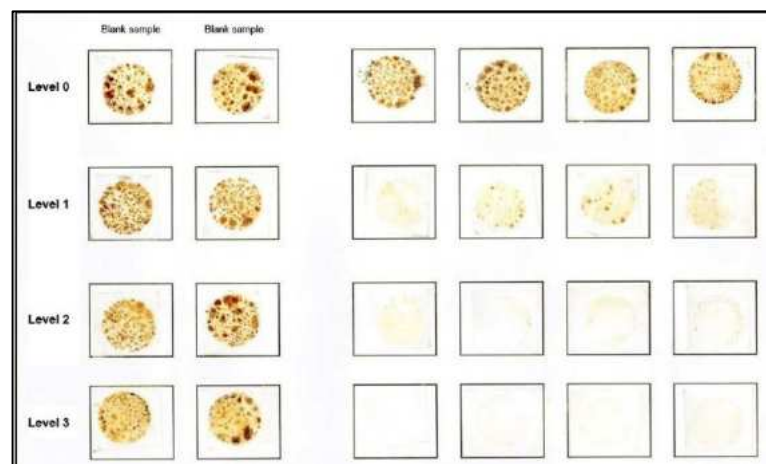


Figure 1: Evaluation of the results of K test

2.3.2 Contact and distance test, preserving jar test (KDW test) acc. VW 50164, subsection 5.2 *)

Test sheets of a steel:

DC 03 (cold-rolled, low-carbon steel, material no. 1.0347 with the dimensions 50 mm × 90 mm × 0.5 mm (Q-Lab Deutschland GmbH) batch: 404151248

Test setup: spacer frames are each with the VCI packaging material
 packaged spacer frames, each with 5 test sheets, 2 packs in a sealed 1-l preserving jar, preserving jar filled with 15ml demin. water



Test cycles: 16 h @ (40±1)°C
8 h @ (23±2)°C
Number of test cycles: maximum 55 cycles
Evaluation: The surface condition of the test sheets is determined as per section 4.7.
The extent of the corrosion is evaluated acc. VW 50164 table 2 on the basis of DIN 51802 by issuing rust grades for the front and back of the test sheet

2.3.3 Humid climate cycle test with model packaging, metal samples in direct contact (KON test), acc. VW 50164, subsection 5.3 *)

Test sheets of a steel:
DC 03 (cold-rolled, low-carbon steel, material no. 1.0347 with the dimensions 50 mm × 90 mm × 0.5 mm (Q-Lab Deutschland GmbH) batch: 404151248
Test setup: separately packaged test sheets
Test cycle: DIN EN 60068-2-30, a 24-h cycle consists of the following stages:
6 h @ 25°C, RH = 98%
3 h heat-up phase from 25 °C to 55 °C @ RH ≈ 95%
9 h @ 55 °C, RH = 93%
3 h cool-down phase from 55 °C to 25 °C @ RH ≈ 98%
3 h @ 25 °C, RH = 98%
Number of test cycles: maximum 40 cycles
Evaluation: The surface condition of the test sheets is determined as per section 4.7.
The extent of the corrosion is evaluated acc. VW 50164 table 2 on the basis of DIN 51802 by issuing rust grades for the front and back of the test sheet

2.2.4 Humid climate cycle test with model packaging, metal samples in spacer frame (DIS test), acc. VW 50164, subsection 5.4 *)

Test sheets of a steel:
DC 03 (cold-rolled, low-carbon steel, material no. 1.0347 with the dimensions 50 mm × 90 mm × 0.5 mm (Q-Lab Deutschland GmbH) batch: 404151248
Test setup: Model packaging using spacer frame
Test cycle: DIN EN 60068-2-30, a 24-h cycle consists of the following stages:
6 h @ 25°C, RH = 98%
3 h heat-up phase from 25 °C to 55 °C @ RH ≈ 95%
9 h @ 55 °C, RH = 93%
3 h cool-down phase from 55 °C to 25 °C @ RH ≈ 98%
3 h @ 25 °C, RH = 98%
Number of test cycles: maximum 40 cycles



Evaluation: The surface condition of the test sheets is determined as per section 4.7. The extent of the corrosion is evaluated acc. VW 50164 table 2 on the basis of DIN 51802 by issuing rust grades for the front and back of the test sheet

2.2.5 Humid climate cycle test with model packaging made of VCI-free outer packaging, enclosed segments of the VCI-emitting packaging material, and metal samples in the spacer frame (DISU test), acc. VW 50164, subsection 5.5^{*)}

Test sheets of a steel:

DC 03 (cold-rolled, low-carbon steel, material no. 1.0347 with the dimensions 50 mm × 90 mm × 0.5 mm (Q-Lab Deutschland GmbH) batch: 404151248

Test setup: Model packaging using spacer frame, using 2 blanks of VCI film

Test cycle: DIN EN 60068-2-30, a 24-h cycle consists of the following stages:

6 h @ 25 °C, RH = 98%

3 h heat-up phase from 25 °C to 55 °C @ RH ≈ 95%

9 h @ 55 °C, RH = 93%

3 h cool-down phase from 55 °C to 25 °C @ RH ≈ 98%

3 h @ 25 °C, RH = 98%

Number of test cycles: maximum 40 cycles

Evaluation: The surface condition of the test sheets is determined as per section 4.7. The extent of the corrosion is evaluated acc. VW 50164 table 2 on the basis of DIN 51802 by issuing rust grades for the front and back of the test sheet

3 Test results

3.1 Test of the association of reference film with VCI film

	LDPE film	VCI film
layer thickness [μm]	168 ± 7	156 ± 8
Ash [mass %]	$0,020 \pm 0,010$	$0,415 \pm 0,013$
FTIR transmittance spectra	Appendix 1	
FTIR transmittance spectra of the ash	Appendix 2	
DSC curves	Appendix 3	

3.2 Analysis for the presence of secondary amines and nitrite according TRGS 615

Content of secondary amines and nitrite in the VCI film; GSO Document TRGS 615: Technical Rules for Hazardous Substances – restrictions on the Use of Anticorrosion Agents whose use can lead to the formation of N-Nitrosamines, Sample delivery (VCI film) from November, 2nd 2015







Compliance with the requirements of TRGS 615 on the restriction of use for corrosion inhibitors, in the use of which N-nitrosamines can occur, has been analyzed analytically. The following Table 2 summarizes the examination method and the measured content for the respective compounds.

Table 2: Analysis on the presence of secondary amines and nitrite according to TRGS 615

Substance name	Applied analysis method	Concentration [%]	Requirements in accordance with TRGS 615	assessment
Nitrite	photometry	0.002	$\leq 0.1 \%$	fulfilled
Morpholine	Gas chromatography and mass spectrometry	< 0.01	$\leq 0.02 \%$	fulfilled
Piperazine	Gas chromatography and mass spectrometry	< 0.01	$\leq 0.02 \%$	fulfilled
Diethanolamine	Gas chromatography and mass spectrometry	< 0.01	$\leq 0.02 \%$	fulfilled
4,4'-Methylene-bis-morpholine	Steam distillation, photometry	< 0.001	$\leq 0.02\%$	fulfilled

3.3 Test of the corrosion protection effect

3.3.1 Flask test, (K test)

	VCI film				LDPE film ≡ reference	
evaluation (level)	3	3	3	3	0	0
						
protection factor SF	3					
rating	good					

3.3.2 Preserving jar test (KDW test)

	VCI film								LDPE film ≡ reference			
<u>1. contact surfaces</u> Failure of contact surfaces after cycles	more than 56								More than 16 (12 for calculation)			
rust grades RN	0	0	0	0	0	0	0	0	0	0	0	
protection factor SF	more than 4,67											
level _{KDW-contact}	3 since SF >4.5											
rating _{KDW-contact}	good											
<u>2. intermediate spaces</u> Failure of intermediate spaces after cycles	more than 56								More than 16 (12 for calculation)			
rust grades RN	0R	0	0	0	0	2	0	0	1	0R	1	0
protection factor SF	more than 4,67											
level _{KDW-inter.}	3 since SF >4.5											
rating _{KDW-inter.}	good											
<u>3. gap</u> Failure of gaps after cycles	more than 56								More than 16 (12 for calculation)			
rust grades RN	1	0	0	0	0	0	0	0	1R	0R	1R	2
protection factor SF	more than 4,67											
level _{KDW gap}	3 since SF >4.5											
rating _{KDW gap}	good											

3.3.3 Metal samples in direct contact (KON test)

	VCI film			LDPE film ≡ reference	
Failure of direct contact after cycles	40 (36 for passed)			14 (8 for calculation)	
rust grades RN	2 / 1R	0 / 1R	1R / 2	1 / 2	
protection factor SF	4.5				
level	2 since SF ≤ 4.5				
rating	moderate				



3.3.4 Metal samples in spacer frame (DIS test)

	VCI film			LDPE film ≡ reference
Failure of spacer frame samples after cycles	more than 40			14 (8 for calculation)
rust grades RN	0 / 0	0 / 0	0R / 0R	1 / 2
protection factor SF	more than 5			
level	3 since SF >4.5			
rating	good			

3.3.5 VCI-free outer packaging, enclosed segments of the VCI-emitting packaging material, and metal samples in the spacer frame (DISU test)

	VCI film			LDPE film ≡ reference
Failure of spacer frame samples after cycles	more than 40			14 (8 for calculation)
rust grades RN	0 / 0	1R / 0	1R / 0	2 / 2
protection factor SF	more than 5			
level	3 since SF >4.5			
rating	good			

3.4 Summary of test results from point 3.3.1 to 3.3.5

Test	protection factor	level	rating	Corrosion protection rating
K test	-	3	good	2
KDW test	> 4.5	3	good	2
<i>contact</i>	> 4,5	3	good	2
<i>intermediate space</i>	> 4,5	3	good	2
<i>gap</i>	> 4,5	3	good	2
KON test	4.5	2	moderate	3
DIS test	> 4.5	3	good	2
DISU test	> 4.5	3	good	2
Total evaluation		2,8	Good, packag- ing material is suitable for overseas transport	2,2



4 Assessment

1. The VCI packaging material does not exceed the permitted limit values for diethanolamine, morpholine, 4,4'-methylene-bis-morpholine, piperazine and nitrite in the finished product.
2. A film thickness of 156 μm was determined.
3. The tests KDW, KON, DIS and DISU were passed with rust grades RN 2 and better.
4. Protection level 2.8 was calculated as the arithmetic mean of tests K-, KDW, KON, DIS and DISU test for the corrosion protection effect. The corrosion protection effect was assessed with good, suitable for overseas transportation.
5. The corrosion protection of the VCI film in the tests K, KDW, KON, DIS and DISU test is graded with 2.2* (best grade 2.0).
(* Average of all the test scores)

The corrosion protection requirements are fulfilled.

Magdeburg, 07.11.2016
Institut für Lacke und Farben Magdeburg gGmbH

Dr. Ute Holzhausen
(Laboratory head)

Dr. Ing. Susanne Bender
(Deputy Laboratory head)

The test results refer only to the subjects of testing. The publication of the results in extracts is subject to the approval of the Institut fuer Lacke und Farben Magdeburg gGmbH.

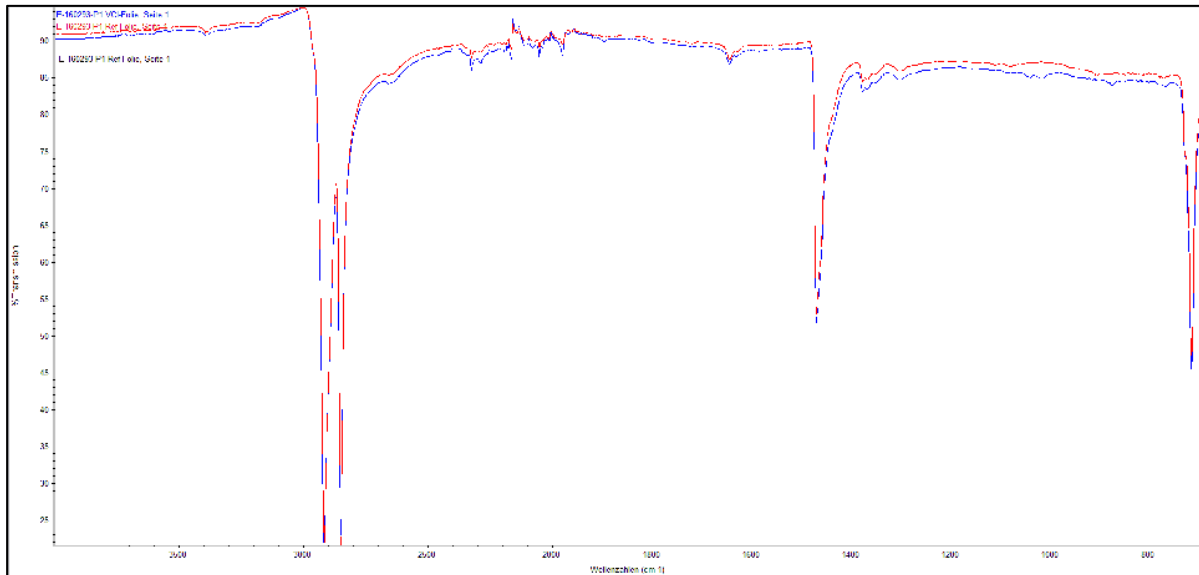


Figure 2: association of LDPE reference film with the VCI film, FTIR transmittance spectra, spectra correlation=98.3%

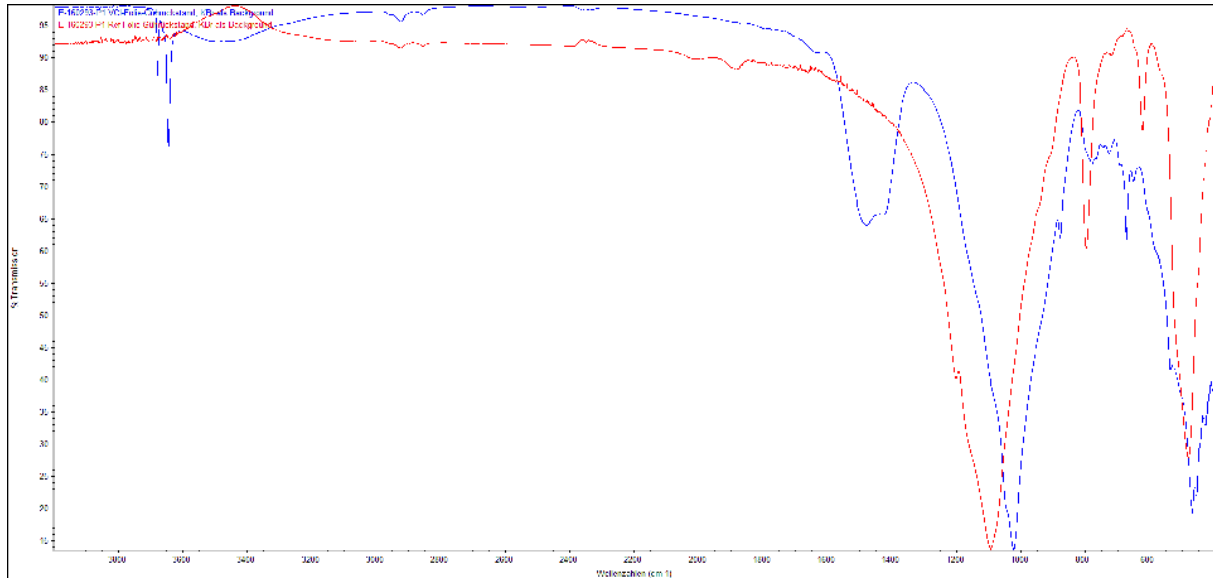


Figure 3: association of LDPE reference film with the VCI film; FTIR transmittance spectra of the ash, spectra correlation=8.9%

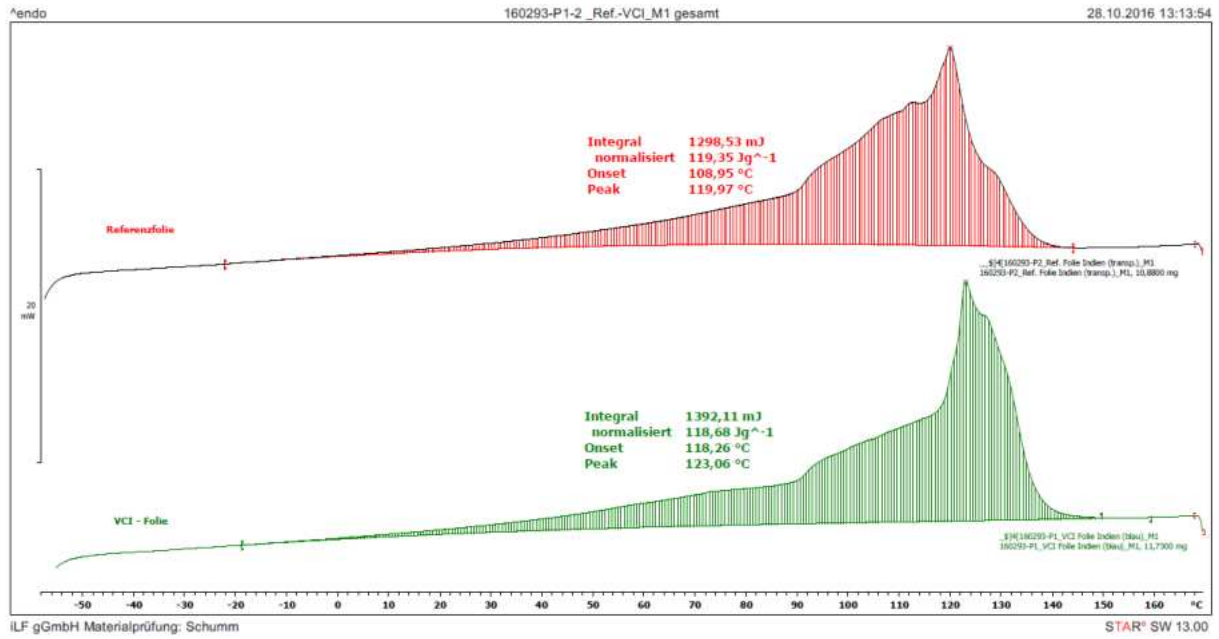


Figure 4: association of LDPE reference film with the VCI film; DSC-curves