



INSTITUT FÜR KORROSIONSSCHUTZ DRESDEN GMBH

Privatwirtschaftliche Forschungsstelle



Beratung - Schadensfallaufklärung - Qualitätssicherung - Forschung - Prüfung

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Untersuchungsbericht

UB400/076/12

Orderer: VCI Brasil Indústria e Comércio de Embalagens Ltda.
Rodovia Marechal Rondon, km 334,3
CEP 17048-690 BAURU/SP (BRASIL)

Date of order: 2012-08-21

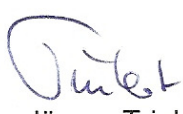
Date of delivery: 2012-08-13

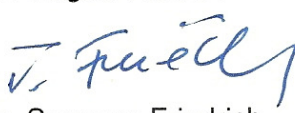
Period of testing: 2012-09 to 2012-12

Order: Test of the correspondence of the VCI film VCI 691 V3,
delivery 2012-08-13, with the requirements of the guideline
corrosion protection by VW AG

Number of order: LA4/248/12/124103, LA4/312/12/124103

Number of pages: 4

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Dresden, 11.12.2012

1 Conceptual Formulation

The conceptual formulation encloses the quantitative determination of the components, declared in the confidential full compositional disclosure of the orderer. Moreover the conformance of the material with the german guideline TRGS 615 had to be examined. For that purpose the total contents of Nitrite and primary, secondary and tertiary Amines in the material are determined quantitatively.

This procedure is performed in accordance with the updated guideline for the use of VCI materials of the VOLKSWAGEN AG Wolfsburg from 2011-05-20 and 2012-10-24.

The orderer delivered two materials for testing:

- **VCI material: film VCI 691 V3 (delivery 2012-08-13)**
- **Reference material: reference film with pyrogenic silica (delivery 2012-10-19).**

2 Methodical Information

Quantitative determination of the components, declared in the confidential full compositional disclosure of the orderer

The two primary / tertiary Amines in the material were determined by cation chromatography (IKS procedure) after a triple ultrasonic extraction of the material with deionized water. An ion chromatograph Dionex ICS 5000 with gradient elution and cation suppression was used.

The Benzoic Acid in the VCI film was determined by anion chromatography (IKS procedure) after a triple ultrasonic extraction of the material with deionized water. An ion chromatograph Dionex ICS 5000 with gradient elution and anion suppression was used.

The 4/5-Methyl-1H-Benzotriazole in the material were determined by another accredited laboratory by a coupling of high performance liquid chromatography and mass spectrometry (HPLC-MS) in a Methanol extract.

The Caprylic Acid in the film was determined by another accredited laboratory by a coupling of gas chromatography and mass spectrometry (GC-MS) in a Methanol extract after esterification.

Conformance of the material with the german guideline TRGS 615 (Secondary Amines and Nitrite)

The Nitrite concentration in the VCI material is determined quantitatively by anion chromatography (according to DIN EN ISO 10304-1: 1995) after a triple ultrasonic extraction of the film with deionized water.

The concentration of secondary Amines in the material is determined quantitatively by cation chromatography (IKS procedure) after a triple ultrasonic extraction of the film with deionized water and by gaschromatography / mass spectrometry.

Comparability of corrosion protection VCI film and his reference material

To verify the comparability of the corrosion protection VCI film and his reference material for the corrosion protection tests the film thickness, ash content of the film and the chemical composition of the ash are determined. The amount of ash residue of the film is determined in accordance with DIN EN ISO 3451 at 600°C. The chemical composition of the ash residue is analyzed by EDX-spectroscopy.

3 Test results

The following test results were determined for the sample material:

Parameter	Dimension	Film VCI 691 V3 (delivery 2012-08-13)
Total content of Nitrite (calculated as NaNO ₂)	wt-% NaNO ₂	< 0,01
<i>Total content of analyzed secondary Amines:</i>		
Diethanolamine	wt-%	< 0,005
Morpholine	wt-%	< 0,005
Dicyclohexylamine	wt-%	< 0,01
Piperazine	wt-%	< 0,01
<i>Total content of the detected primary and tertiary Amines:</i>		
3-Dimethylaminopropylamin CAS-No.: 109-55-7	wt-%	0,026
2,2'-(Methylimino)diethanol CAS-No.: 105-59-9	wt-%	not detected *
<i>Another components:</i>		
Benzoic Acid	wt-%	0,03
4-Methyl-1H-Benzotriazol	wt-%	0,004
5-Methyl-1H-Benzotriazol	wt-%	0,005
Caprylic Acid	wt-%	0,12

The figures < 0,005 and < 0,01 mean, that the content of the component is smaller than the minimum determination value of the analytical method.

** After addition of the standard substance 2,2'-(Methylimino)diethanol (CAS 105-59-9) directly to the eluate of the VCI film the chromatogram shows two peaks side on side on the place, where the standard substance eluates. That means the substance in the VCI material can not be exactly 2,2'-(Methylimino)diethanol. The orderer / producer of the VCI film by request declared in writing that the used substance for producing the VCI film is CAS 105-59-9, obtained from BASF AG in Germany with a pureness > 99 %.*

Apparently the declared amin reacts in the masterbatch or in the film (extrusion at $T > 200^{\circ}\text{C}$) and forms a slightly modified amin.

The film thickness, the amount of ash residue and the chemical composition of the ash of the analyzed corrosion protection film VCI 691 V3 (delivery 2012-08-13) and his reference material corresponds with each other in predefined limits.

4 Conclusion

The analyzed VCI film

VCI 691 V3 (delivery 2012-08-13)

shows:

1. No secondary Amines (TRGS 615)
2. A Nitrite concentration (calculated as NaNO_2) smaller 1,0 Ma-% (TRGS 615)
3. The analyzed chemical composition of the assayed VCI material corresponds with the declared formulation of the confidential full compositional disclosure of the orderer.

Therefore the sample material comply the requirements of the guideline for the use of VCI materials, instruction procedure to the check of VCI materials of the VOLKSWAGEN AG Wolfsburg, last updated on 2012-10-24.

The analyzed VCI material comply also the requirements of the german TRGS 615 (2007).