



Beratung - Schadensfallaufklärung - Qualitätssicherung - Forschung - Prüfung

- Akkreditiertes Prüflabor für Korrosion, Korrosionsschutz und Korrosionsanalytik
- DAkkS-Registrierungsnummer: D-PL-19138-01-00
- Institut im Verbund der Technischen Akademie Wuppertal e. V.
- Institut an der TU Bergakademie Freiberg

☎ 0351 871 7100  
Fax 0351 871 7150

Institut für Korrosionsschutz Dresden GmbH • Gostritzer Str. 65 • 01217 Dresden

## Test Report PB200/014/22\_1

Client: MetPro Verpackungs-Service GmbH  
An der Linde 21  
04838 Jesewitz  
GERMANY

Day of order: 2022-03-10

Receipt of test specimens: 2022-03-09


Period of testing: 2022-03-11 to 2022-06-29

Order: Analysis of "BioCor© 300 RP" 80 µm VCI film according to VW50164



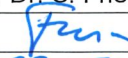
Laboratory job No.: LA2/44/22/222039, LA4/100/22/222039

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Person in charge:   
Dr. Frank Hoffmann

Head of Department /  
Managing Director:   
Dr. Susanne Friedrich

Dresden, 2022-12-22

Prepared: Dr. F. Hoffmann	Checked: Dr. S. Friedrich	Approved: Dr. S. Friedrich
Sign: 	Sign: 	Sign: 
Date: 2022-12-22	Date: 2022-12-22	Date: 2022-12-22

This test report replaces test report PB200/014/22 dated 2022-06-29, which is hereby invalidated.

The publication of test reports in extracts, the reference to the tests for the purpose of promotion and the application of the content of the test report require a written consent of the Institut für Korrosionsschutz Dresden GmbH in every single case. In the case of no other agreement we reserve to dispose of the samples three month after delivery. The statements refer to the test object exclusively.

## 1 Subject

The active agents of the VCI material "BioCor© 300 RP" 80 µm VCI film, which are listed in the confidential declaration of the complete composition by the client, must be determined quantitatively. In addition, the conformity with the German guideline TRGS 615 has to be checked. For that purpose, the total contents of nitrite and secondary amines in the material are to be determined quantitatively.

This procedure is performed in accordance to the standard VW50164, issue 2013-06, "Method for Testing and Releasing VCI Packaging Materials" of the Volkswagen AG Wolfsburg.

This standard requires verification of the following points:

- Analytical verification of compliance with the requirements of TRGS 615 and further requirements of VW AG: no secondary amines and a sodium nitrite content  $c(\text{NaNO}_2) \leq 1.0$  % by mass (recommendation TRGS 615:  $c(\text{NaNO}_2) \leq 0.5$  Ma -%)
- Quantitative determination of the components of the disclosed formula as a basis for comparison for subsequent control determinations from current deliveries to Volkswagen AG
- Comparability of film thickness and ash content of reference film and VCI film

## 2 Supplied Materials

The following materials were handed over to the IKS by the client for these investigations:

- (1) **Corrosion protection film "BioCor© 300 RP", 80 µm (sample receipt: 2022-03-09)<sup>1</sup>**
- (2) **Associated VCI-free Reference film of the same name, 80 µm (sample receipt: 2022-03-09)**

## 3 Analysis methods

### Quantitative determination of the declared active ingredients in the VCI material<sup>2</sup>

- Sodium Benzoate, Nonanoic Acid and Potassium Sorbate in the film were determined by a coupling of gas chromatography and mass spectrometry (GC-MS) in a methanol-extract after esterification (methylester) according to standard DIN EN ISO 12966-2:2011-05.

### Conformity of the material with the German guideline TRGS 615 (secondary amines and nitrite)

- The nitrite concentration in the VCI film was determined quantitatively by anion chromatography (according to DIN EN ISO 10304-1:2009-07) after 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 triple ultrasonic extraction of the material with deionized water and by gas chromatography / mass spectrometry.

### Comparability of corrosion protection VCI film and its reference material

To check the association of a reference film with the VCI film to be tested, of both the layer thickness (10-fold determination) and the ash (3-fold determination) in mass % after calcination at 600 °C as per DIN EN ISO 3451-1 were determined, and EDX spectra of the ash were recorded.

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<sup>1</sup> At the time of testing, the film was designated as "BIOCOR©-R". The name was later changed to "BioCor© 300 RP".

<sup>2</sup> Not within the accredited scope

**Utilized accredited testing instruments (TI):**

The DIONEX ICS 5000 ion chromatograph from DIONEX Corporation (TI card no. PMK 400-14), the automatic pipettes from Eppendorf AG (TI card no. PMK 400-12), the calibrated precision balance LA 230 P-OEC from Sartorius AG Goettingen (TI card no. PMK 400-3) and the laboratory refrigerator (TI-card-No. PMK 400-19) were used for the analyses.

For the ash content determination according to DIN EN ISO 3451-1 were used the muffle furnace CWF 11/13 of Carbolite GmbH (TI-card-No. PMK 200-18) and the calibrated precision balance LA 230 P-OEC of Sartorius AG Goettingen (TI-card-No. PMK 400-3).

The coating thickness measurement instrument Deltascope of Helmut Fischer GmbH (PMK 300-11.4) was used to determine the film thickness.

## 4 Results

The following analysis results (Table 1) were obtained for the materials (the substance concentrations are mean values of duplicate determinations):

Table 1: Analysis results

Parameter	Dimension	Corrosion protection film "BioCor® 300 RP", 80 µm (sample receipt: 2022-03-09)	Associated VCI-free Reference film of the same name, 80 µm (sample receipt: 2022-03-09)
Layer thickness ± expanded uncertainty	µm	77.4 ± 3.0	78.8 ± 7.8
Ash content ± expanded uncertainty	wt-%	1.036 ± 0.065	0.854 ± 0.057
Nitrite calculated as NaNO <sub>2</sub> ± expanded uncertainty	wt-% NaNO <sub>2</sub>	< 0.0005	-
<i>Secondary amines<sup>1</sup></i>			
Diethanolamine	wt-%	< 0.001	-
Morpholine	wt-%	< 0.005	-
Dicyclohexylamine	wt-%	< 0.01	-
Piperazine	wt-%	< 0.01	-
<i>Other declared components<sup>1</sup></i>			
Sodium Benzoate calculated as Benzoate	wt-%	<b>0.034 (GC-MS)</b>	-
Nonanoic Acid	wt-%	<b>0.071 (GC-MS)</b>	-
Potassium Sorbate calculated as Sorbate	wt-%	<b>0.003 (GC-MS)</b>	-
<i>Other ingredients found<sup>1</sup></i>			
Octanoic Acid	wt-%	0.007 (GC-MS) <sup>2</sup>	-
Decanoic Acid	wt-%	< 0.004 (GC-MS) <sup>2</sup>	-

The figures < 0.005, < 0.001 and < 0.01 etc. mean that the content of the component is smaller than the minimum detection limit of the analytical method. n.d. = component not determinable

The eluates were each prepared 30 min at 60 °C ± 4 °C in an ultrasonic bath and filtered with polyethersulfone syringe filters (0.45 µm) prior to measurement.

No secondary amines could be detected by headspace GC-MS.

<sup>1</sup> not within accredited scope

<sup>2</sup> Decanoic Acid was detected qualitatively. These small amounts of Octanoic and Decanoic Acid are probably impurities of Nonanoic Acid.

### **Measurement uncertainty**

The expanded uncertainty of measurement is given, which results from the standard uncertainty of measurement multiplied by the coverage factor  $k = 2$ . The value of the measurand lies within the assigned value interval with a probability of approximately 95 %.

## **5 Requirements**

### **VCI material**

The confidential disclosed chemical composition of the active agents in the VCI material has to correspond with the analyzed chemical composition of the VCI material sample. VCI materials that do not conform to the declaration will be rejected without testing the VCI effect.

Furthermore, the presence of secondary amines and nitrite has to be examined (basis of the analysis is TRGS 615).

According to VW50164 section 4.1, for a release of the VCI material, diethanolamine, morpholine, and piperazine must not exceed a mass fraction of 0.02 %. The presence of any secondary amines is also not permitted by VW.

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 to Volkswagen Central Group Quality Assurance for Logistics and CKD (K-GQZ-L) in writing that the VCI material cannot form or release any N-nitrosamines when used properly.

### **Reference material**

According to VW50164 section 4.2, a prerequisite for the evaluation of the tests described in section 5 is that clear signs of corrosion are produced on the specimen in the same test setup as for testing the VCI material using segments of a similar but VCI-free packaging material (paper of the same grammage, LDPE films of the same layer thickness and type of production, monoextrusion or coextrusion) under the same test conditions within a limited time.

The association of a reference film with the VCI film to be tested must be ensured in advance by means of the following comparative analyses of both materials:

- Determination of the layer thicknesses
- Determination of the ash in mass % (after calcination at 600 °C) as per DIN EN ISO 3451-1  
The fraction of inorganic VCI components and their conversion products must be deducted from the determined ash of the VCI film.
- Recording of EDX spectra of the ashes

## 6 Conformity

The analyzed VCI material

### **(1) Corrosion protection film “BioCor© 300 RP”, 80 µm (sample receipt: 2022-03-09)**

contains:

1. No secondary amines (TRGS 615)
2. No nitrite (calculated as NaNO<sub>2</sub>) (TRGS 615)

Analytical findings IKS:            Secondary amines below detection limit (< 0.01 % by mass)  
    Nitrite concentration (calculated as NaNO<sub>2</sub>) below detection limit  
    (< 0.0005 wt-%)

Thus, the material (1) meets the requirements of TRGS 615 (2007).

The IKS analysis results of (1) are qualitatively consistent with the client’s disclosed chemical composition. It is likely that only a fraction of the active ingredients could be extracted from the VCI film.

The thickness values of the reference film (2) and the corresponding VCI film (1) are comparable. The difference in the ash contents of the reference film (2) and the VCI film (1) only comes from declared VCI ingredients. The test of the corrosion protection effect with this reference film is permissible.

The VCI film sample (1) “BioCor© 300 RP”, 80 µm (sample receipt: 2022-03-09) and the associated VCI-free Reference film of the same name, 80 µm (sample receipt: 2022-03-09) thus meet the analytical requirements for release by Volkswagen AG Wolfsburg listed in paragraph 5.

**Notice:**

The conformity statement refers only to the above requirements.

The following decision rule(s) were applied for the conformity assessment(s):

Measurement uncertainty was considered as follows:

The test result including the expanded measurement uncertainty (coverage factor k=2, coverage probability 95%) complies with the standard/specification.

## 7 Used accredited standards and regulations

Table 2: used accredited standards and regulations

standard / regulation	edition
VW50164	2013-06
DIN EN ISO 3451-1	2008-11
DIN EN ISO 10304-1	2009-07