

# CHALMERS



## Method for measuring degreasing on different substrates

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**Bachelor of Science Thesis**

Department of Chemical and Biological Engineering

*Division of Applied Chemistry*

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## **Preface**

This thesis is the final of my Bachelor of Science degree in Chemistry Engineering at the department of Chemical and Biological Engineering at Chalmers University of Technology (CTH). The thesis has been carried out at Surface Treatment Engineering on the department (93662) at Volvo Car Corporation (VCC) in Gothenburg, Sweden. The work was performed during the period September to November 2010.

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Gothenburg, November 2010

Renée Serenban

## Abstract

Several metal parts from the car body are covered with oil when delivered to VCC. The oil is important to remove in the pre-treatment process; otherwise, there could be problems in the following steps such as disturbances in the phosphate layer. This in turn can lead to poorer adhesion, corrosion, and painting defects.

This thesis is to try to develop a test method to measure degreasing of oils and which should give similar results as in production process at VCC.

The experimental procedure is mainly based on guidelines from Chemetall and from the report ER-513 386, *Test of new degreaser, Gardoclean S5193A, from Chemetall*. Both tests are performed in the same way and the preparations. Results given in this report are compared with results from previous report ER-513 386 to see if repeated trials give the same result. In most cases, they do. Some, however, differ.

The method was also compared to production. Test panels went through the pre-treatment process followed by a visual assessment of the phosphate layer. Most panels were good but some of them had spots on their surface. Investigation by SEM only showed minor disturbances. However, the results from the laboratory method were not always consistent with the results from the production process.

Subsequent steps in the experiment were to vary the parameters temperature and concentration, to achieve an improvement when increasing these parameters. Since not all of the oils performed better the method is not a general solution, instead it can be used for guidance and as a reference for future experiments. Each of the oil should be considered and judged individually.

Not all of the compounds and information is declared in the data sheets of the oils, the rest of the information is classified. With this vague information, it is difficult to make a correlation between the oils.

IR and measurement of contact angle have been applied in order to see a link or to understand the relationship of the various oils used in the experiment. However, no connections were found, and other tools are needed to get more information. Due to some uncertainties with the method, contact angle is still interesting to include in further investigation.

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## List of Abbreviations

HDG	Hot Dip Galvanized steel,
CRS	Cold Rolled Steel
EG	Electro Galvanized Steel
GA	Galvannealed
VCC	Volvo Car Corporation
VCS	Volvo Car Corporation Standard
SEM	Scanning Electron Microscope
IR	Infrared
GC-MS	Gas chromatography- Mass spectrometry

# **1 Introduction**

## **1.1 Background**

The experiment is to develop a laboratory method to measure degreasing of oils for several oil combinations on different metal substrates. Suppliers deliver metal parts to Volvo Car Corporation, these parts has oil coated on them. The report and the experiment is to find a laboratory method that gives good correlation to the production process regardless of what kind of oil is to be degreased.

## **1.2 Purpose**

The purpose of this thesis is to develop a method for measuring degreasing of different types of substrate and oil combinations. Degreasing in laboratory sometimes does not give the same result as in production. In addition, the result in laboratory should give the same result as in production.

Degreasing is important for subsequent steps in the process for the car body to provide a protective phosphate coating, good adhesion, and good painting.

## **1.3 Method**

The work is mainly based on experiments. Guidelines are designed by one of VCC suppliers, Chemetall, for the experiment how the method can be performed. The task is to adjust the parameters to obtain better results.

## **1.4 Constraints**

Due to the limit of ten weeks of full-time work, the amount of test objects is restricted to approximately twelve oils. The limitations to this experiment, is only to vary the parameters: temperature and concentration.

## 2 Pre-treatment process

In the production car bodies is entering a twelve-step process containing degreasing, rinsing, activation, phosphating, and passivation. To make this possible all oil and grease on the metal surface must be removed by degreasing in order to obtain good activation, phosphating, and passivation.

The phosphating layer serves as a corrosion protection and as a pre-treatment process for painting.

**Step 1:** is an alkaline *high pressure degreasing* step where pollutants and particles are washed off.

**Step 2:** an alkaline *spray degreasing* step where the body is mainly washed on the outside

**Step 3:** an alkaline *dip degreasing*, and it is especially the inside of the car body that is washed

**Step 4:** is a *spray rinse* step with city water, and

**Step 5:** is a *dip rinse* step also with city water and removes the last traces of the degreasing.

When the body has passed degreasing- and rinsing steps, the metal surface is virtually free from pollution.

**Step 6:** dipping the car body into the *activation step* so the entire body is activated. It increases the number of crystal sprout, so that a well-fitting crystal layer in the phosphate step is obtained. Amount of phosphate crystals, coating layer and the processing time is effected by these crystal sprouts on the metal surface.

**Step 7:** Next step in process, the *phosphating step* where the crystal layer is formed. A phosphating bath is a water solution with phosphoric acid and some metal ions e.g. zinc and manganese. Other additives are oxidizing agents (nitrate, nitrite, chlorate etc), nickel, fluorine etc

**Step 8:** is a *spray rinse* step where the car body is sprayed with deionised water and

**Step 9:** a *dip rinsing* in deionised water.

**Step 10:** The phosphate-coated surface must be sealed in the passivation step. It occurs when the body is dipped in a zirconium solution and any pores at the surface are sealed. The passivation improves paint adherence and prevents blistering.

**Step 11:** is a *spray rinse* step where deionised water is used and

**Step 12:** a *dip rinse* step also with deionised water [2] [3]

### 3 Experiment

The materials tested in this study are substrates, different kind of oils and degreaser. Same oils, which are tested in laboratory, are also tested through the process to see and compare the result with each other.

#### 3.1 Substrate used in method

HDG, CRS, EG and GA are used as substrate in experiment. These substrates are sheet metals used on cars in reality.

#### 3.2 Oils used in method

There are two different kinds of oils, mineral and vegetable oils.

Mineral oils originally come from crude oil and do not contain any minerals, vitamins, and no fatty acids. Oil is viscous to less viscous and insoluble in water (*except for emulsion oils*).

Vegetable oils are formed in the building life process in plants. A disadvantage of vegetable oils is that they are in comparison with mineral oils are more expensive as raw materials. [19]

Mineral oils;

- QuakerDraw 362
- Aral Ropa W35 (emulsion oil)
- Iloform PL6
- Aral Ropa 4093
- Multidraw KTL N16
- SW4020
- Anticorit RP 4107S
- Umformöl 60

Vegetable oils;

- Binol Cut 10
- Binol Cut 20

Name of the oil	Oil type	Additives	Viscosity	Density
<b>Draw 362</b>	Mineral oil	1-5% calcium sulfonate	362 mm <sup>2</sup> /s at 40 °C	0.925 g/cm <sup>3</sup> at 15°C
<b>Aral Ropa W35</b>	Mineral oil	20-50% sodium sulfonate 10-20% alcohol etoxylate		
<b>Iloform PL6</b>	Mineral oil	8 % ester oil 3% calcium sulfonate 1,3% zinc-based	<i>No data found</i>	<i>No data found</i>

		antiwearadditive		
<b>Aral Ropa 4093</b>	Mineral oil	1-10% polyisobutylene 1-10% fattyacidester 1-10% metalcarboxylate 1-10% sodium sulfonate	50-60 mm <sup>2</sup> /s at 20°C	0,86 g/cm <sup>3</sup> at 20°C
<b>Multidraw KTL N16</b>	Mineral oil	1-2,5% polysulphide	<i>No data found</i>	<i>No data found</i>
<b>SW4020</b>	Mineral oil	8 % ester oil 0,5 % antioxidant 91,5 % synthetic oil	<i>No data found</i>	<i>No data found</i>
<b>Anticorit RP 4107S</b>	Mineral oil		145 mm <sup>2</sup> /s at 20°C	0,887 g/cm <sup>3</sup> at 15°C
<b>Binol Cut 10</b>	Vegetable oil	5-10% alkanolamines fattyacidester < 5% monoisopropanolamin 2-5% oxazolidin	<i>No data found</i>	<i>No data found</i>
<b>Binol Cut 20</b>	Vegetable oil	<i>Non components occurring data sheet</i>	<i>No data found</i>	<i>No data found</i>
<b>Umformöl 60</b>	Mineral oil		<i>No data found</i>	<i>No data found</i>

*Table 1.* Data table for oils used in test [6]-[15]

All content of compounds in the oils is not declared in the data sheets; rest of the information is classified and will not be handed out from the suppliers.

### 3.3 Degreaser used in method

The degreasing agent Gardoclean 855/1 is used in laboratory during the test methods also used in the pre-treatment process at VCC.

#### Gardoclean 855/1 contains:

- 40 % disodiumtetraborate pentahydrate
- 12, 5 % pentasodium triphosphate
- 11 % sodium hydroxide
- 10, 5 % sodium metasilicate pentahydrate
- 10 % trisodiumphosphate
- 10 % tetra sodium pyrophosphate
- 6 % Fatty alcoholalkoxylat

Content of Gardoclean 855/1 is no different from other degreasing agents. It contains substances such as salts, surfactant, phosphate etc. These compounds give a corrosion protection and high pH, which is wanted in a degreaser agent. [16]

## 4 Methods

The method is based on guidelines from Chemetall. The test method shall be effective, pose no greater cost, uncomplicated to accomplish and also obtain a similar result as the pre-treatment process is giving. It is only two parameters that have been varied in this test; the temperature and the concentration.

### 4.1.1 Preparation with oil of the metal sheets

All specimens were first given a code for easy identification during preparation and during the process. Six test panels were prepared for using in laboratory and three panels in production.

The test panels delivered to VCC contain an oil coating. The oil coating is first removed in a heptane bath and after rinsed with acetone. Afterwards the test panels are weighed, *see Appendix I*. A new layer of oil, which is to be tested, is applied with a brush on the substrate. The panels are allowed to dry for 24 hours and afterwards the panels are measured again by weighing, to see how much oil there is left on the substrate. The test panels are cut in half in a measure of 100\*100mm, the test method begins. (*See Standard VCS 1029,639(2) alternative B, with deviation for application of the oil with a brush on the substrate instead of dipping.*)

### 4.1.2 Degreasing test

1: Degreasing bath containing degreaser Gardoclean 855 / 1 is placed on a hot plate magnetic stirrer in a beaker with a volume of 2500 ml. The selected temperature which should be achieved is set.

2: Panel immersed in the beaker with the degreasing agent and stirred around for 30 seconds.

3: The panel is lifted out of the degreasing bath and it is followed by a drying time of 10 seconds in which the panel is held in a vertical position, so little of the amount of degreaser and oil may run back to the beaker again.

4: The panel is dipped and stirred for 30 seconds in subsequent beaker containing only deionized water which is placed on a hot plate magnetic stirrer with a temperature of 30 degrees.

5: After rinsing the panel in the beaker with deionized water the panel is lifted up and held in a vertical position for 10 seconds.

6: Remaining water film is judged visually in a scale of 0-100 percentage. Step 1-6 is one cycle and is repeated at least three times and up to five times if not a good result is achieved.

For example the water film is 100 percent covered on the substrate, means that the degreasing are successful and that all oil have been removed. [4] [5]



*Fig. 1* Picture of the test arrangement

### 4.1.3 Other methods

#### Contact angle

The contact angle is the angle at which the oil interface meets the solid surface. It is a method of calculating contact angles based on the diameter of sessile drops. By making this approximation it is possible to calculate the contact angle, by measuring the drop diameter of the oil, 10  $\mu$ l. Diameter was measured of the drop directly after it were dropped on the substrate and repeated at least two times. Contact angle can read by in the article *Phagocytosis and Hydrophobicity: A method of calculating contact angles based on the diameter of sessile drops.* [1].

To calculate the contact angle was to see however there are connections between the different oils.

#### IR Spectroscopy

Infrared (IR) spectroscopy is one of the most common spectroscopic techniques used by organic chemists. IR is used both to gather information about the chemistry of a compound and as an analytical tool to assess the purity of a compound.

In IR spectroscopy, an organic molecule is exposed to infrared radiation. When the radiant energy matches the energy of a specific molecular vibration, absorption occurs. Different types of bonds, and thus different functional groups, absorb infrared radiation of different wavelengths are shown in spectra. The wave number is plotted on the X-axis and the percent transmittance is plotted on the Y-axis. [17]

The oils Draw 362, Multidraw KTL N16 and Iloform PL6 were run with an IR analysis to see if a connection will show. Draw 362 and Iloform PL6 have calcium sulfonate as a common denominator, still they give a different result from the degreasing test, therefore they were chosen. Multidraw KTL N16 was chosen randomly.

#### SEM

The scanning electron microscope, or SEM, is an incredible tool for seeing the unseen. SEM is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons.

SEM used in this experiment is to see if the test panels' crystalline structure is disturbed and if they fulfill the requirements of VCS, from when they went through the production. If the crystalline structure is disturbed, it means that the phosphating coat has not 100-percentage coverage and bigger crystals. This can lead to a not so good corrosion resistance and adhesion.

The size of the crystals are up to 10  $\mu$ m for the substrates HDG and CRS, and up to 15  $\mu$ m for GA, these measurement are guiding values for a phosphate layer of good quality. (*See standard VCS 5742, 1049*) [18]

## 5 Results

In the following chapter, where the results will be treated, the current attempts against previous result from the report *ER-513386 "Test of new degreaser, Gardoclean S5193A, from Chemetall"*, is shown and also the results from comparison with production and when parameters are varied.

### 5.1 Comparison test

To demonstrate that equivalent result is obtained with a repeated test, same parameters were used in both of the tests, temperature 50 degrees and concentration of 10g/l. These parameters represent the same ones as used in production. The current experimental values are given on the left side of the table marked with a border and from previous test on right hand side. The results from the cycles are written in percentage of the water film and the amount of oil on each test panel in g/m<sup>2</sup>.

In the report ER-513386, an average amount of oil is listed of how much oil was applied on the test panels. [5]

Oil/Substrate	Amount of oil (g/m <sup>2</sup> )	Cycle 1 (%)	2 (%)	3 (%)	4 (%)	5 (%)	Amount of oil (g/m <sup>2</sup> )	1 (%)	2 (%)	3 (%)	4 (%)	5 (%)
<b>Draw 362/HDG</b>												
Panel 1	9,3	100	100	100				99	100			
Panel 2	9,8	100	100	100			5,3	90	95	99		
Panel 3	9,7	100	100	100				95	95	95		
<b>Aral Ropa W35/HDG</b>												
Panel 1	29,8	100	100	100				100				
Panel 2	32,1	100	100	100			1,2	99				
Panel 3	30,0	100	100	100				100				
<b>Iloform PL6/HDG</b>												
Panel 2	1,2	5	5	5	5	5		10	10	10	20	15
Panel 5	1,1	5	5	5			0,6	15	10	15		
<b>Aral Ropa 4093/HDG</b>												
Panel 6	2,0	10	10	10	20	20		10	10	10	10	20
Panel 3	2,1	15	15	30	30		2,5	10	0	5	5	5
<b>MultidrawKTLN16/HDG</b>												
Panel 3	3,7	65	65	65	80	80		5	10	15	15	10
Panel 1	2,1	60	50	50	55	55	3,2	0	5	10	15	15
<b>AnticoritRP4107S/HDG</b>												
Panel 1	2,8	60	80	80	85	85		25	50	20		
Panel 3	3,3	40	60	60	70	80	7,8	40	45	50		
<b>Umformöl 60/HDG</b>												
Panel 1	1,8	5	10	10	10	10		10	10	10	10	10
Panel 2	1,6	5	5	10			3,0	10	10	10		
<b>SW4020/EG</b>												
Panel 5	2,2	5	5	5	5	5		70	80	85		
Panel 6	2,0	5	5	10			1,4	65	70	65	70	

							80	65	80	80		
<b>SW4020/CRS</b>												
Panel 2	2,3	5	5	10			10	20	70	100	100	
Panel 1	1,4	5	5	10	30	40	1,0	5	5	15	50	
								10	10	15	15	
<b>Binol Cut 20/GA</b>												
Panel 1	2,8	15	20	30	30	30		25	25	25	25	
Panel 3	2,8	10	20	25	30	35	1,9	25	20	25	30	
								40	30	30	35	

**Table 2.** Result from experiment and also the result from the report ER-513386 [5] while using the temperature 50 degrees and the concentration 10 g/l.

Majority of the trials by the different oils gave similar results as the test round before. The oils which not gave the same result were Multidraw KTL N16/HDG and SW4020 on substrate EG and CRS. They did not obtain the same good result like before except for Multidraw KTL N16 who showed a reverse result.

## 5.2 Comparison test result vs. production

To compare the results obtained in laboratory, panels must also be run through the production to see if the similar result were given or to see a connection such as if oils with poor results in the degreasing test could be related to disturbances in the phosphated layer. Some of the phosphate panels from the pre-treatment process came out with spots on their surface.

Panels marked with crosses were also evaluated using the method SEM.

Oil/Substrate	Visual OK/NOK/Observation	Analyzed with SEM	Remark SEM
<b>Draw 362 /HDG</b>			
Panel 1	OK	X	-
Panel 2	OK		
Panel 3	OK		
<b>Aral Ropa W35/HDG</b>			
Panel 1	OK		
Panel 2	OK	X	-
Panel 3	OK	X	-
<b>Iloform PL6/HDG</b>			
Panel 1.1	OK	X	-
Panel 2.1	OK	X	-
Panel 3.1	OK		
Panel 1.2	OK	X	-
Panel 2.2	OK	X	-
Panel 3.2	OK		
<b>Aral Ropa 4093/HDG</b>			
Panel 1	Little spotted	X	-
Panel 2	OK		
Panel 3	OK	X	-
<b>Multidraw KTL N16/HDG</b>			
Panel 1.1	Spotted	X	-
Panel 2.1	Very spotted	X	Flat crystals
Panel 3.1	Spotted		

Panel 1.2	OK	X	-
Panel 2.2	Little spotted	X	-
Panel 3.2	OK		
<b>Anticorit RP 4107S/HDG</b>			
Panel 1	OK	X	-
Panel 2	OK	X	-
Panel 3	OK		
<b>Umformöl 60/HDG</b>			
Panel 1	Spotted + white spots	X	-
Panel 2	Spotted + white spots		
Panel 3	Spotted + white spots	X	-
<b>SW4020/EG</b>			
Panel 1	Little spotted		
Panel 2	Little spotted	X	-
Panel 3	Little spotted	X	-
<b>SW4020/CRS</b>			
Panel 1	OK	X	-
Panel 2	OK	X	-
Panel 3	OK		
<b>SW4020/HDG</b>			
Panel 1	OK	X	-
Panel 2	Spot in corner	X	Flat and bigger crystals
Panel 3	OK		
<b>Binol Cut 10/HDG</b>			
Panel 1	OK		
Panel 2	Little spotted	X	Flat and bigger crystals
Panel 3	Little spotted	X	Flat and bigger crystals
<b>Binol Cut 20/GA</b>			
Panel 1	Little spotted	X	Flat and bigger crystals
Panel 2	Little spotted		
Panel 3	Little spotted	X	Flat and bigger crystals

**Table 3.** Table of visual observation of the plates passing the production.  
Flat – tendency of lying crystals

Some of the tested oils obtained same result in laboratory as in production. Like Draw 362 for example gave a very good degreasing result in laboratory and a good one in production with a phosphated layer without remarks.

SW4020/HDG, SW4020/EG, Binol Cut 10/HDG and Binol Cut 20/GA has received remarks from production. SW4020/EG and Binol Cut 10/HDG have also received a low percentage results in laboratory.

Binol Cut 20/GA obtained good result in laboratory but was visually spotted from production. The substrate galvanized does not react in the same way as the other substrates, size and

shape of crystals are in general different, and that can be a contributing factor to the remarks from the production.



**Fig. 2** Picture of some of the panels that went through the phosphating process. The stripes on the panels are from the production and are not caused by the degreasing test. See Appendix 3 & 4.

### 5.3 Test with various parameters

Third experiment was to test whether the degreasing could be improved by increase of the temperature up to 65 degrees and concentration up to 12,5 g/l. The method itself, in addition to the changes of parameters, is the same as in the first experiment and performed exactly the same way. [4][5]

Oil/Substrate	Nr.	Cycle 1	2	3	4	5
<b>Draw 362 /HDG</b>						
T=50, C=10g/l	1	100	100	100		
T=50, C=10g/l	2	100	100	100		
T=50, C=10g/l	3	100	100	100		
T=60, C=10g/l	1	100	100	100		
T=60, C=10g/l	2	100	100	100		
T=60, C=10g/l	3	100	100	100		
<b>Aral Ropa W35/HDG</b>						
T=50, C=10g/l	1	100	100	100		
T=50, C=10g/l	2	100	100	100		
T=50, C=10g/l	3	100	100	100		
<b>Iloform PL6/HDG</b>						
T=50, C=10g/l	2	5	5	5	5	5
T=50, C=10g/l	5	5	5	5		
T=55, C=10g/l	1	20	20	20	20	25
T=55, C=10g/l	3	20	25	25		
T=60, C=10g/l	4	25	15	15	15	
T=60, C=10g/l	3	25	30	30	30	30
T=60, C=10g/l	1.1	15	20	20	20	25
T=60, C=10g/l	2.1	20	20	20	30	30
T=60, C=10g/l	3.1	15	20	20	30	30
T=65, C=10g/l	6	40	35	35	35	45
T=65, C=10g/l	1	50	45	50	45	45
T=50, C=12,5g/l	6	30	20	25	25	
T=50, C=12,5g/l	4	25	40	40	40	
T=50, C=12,5g/l	1.2	5	15	20	20	20
T=50, C=12,5g/l	2.2	10	10	15	20	20
T=50, C=12,5g/l	3.2	10	15	15	15	15
T=65, C=12,5g/l	2	20	25	30	40	40
T=65, C=12,5g/l	5	30	40	30	45	

**Aral Ropa 4093/HDG**

T=50, C=10g/l	6	10	10	10	20	20
T=50, C=10g/l	3	15	15	30	30	
T=55, C=10g/l	1	20	20	30	30	
T=55, C=10g/l	2	10	10	10	10	
T=60, C=10g/l	5	10	10	15	25	20
T=60, C=10g/l	2	10	10	10	10	
T=65, C=10g/l	4	10	15	15	15	20
T=65, C=10g/l	5	10	10	10	10	10
T=50, C=12,5g/l	4	10	10	10	10	
T=50, C=12,5g/l	1	15	10	10		
T=55, C=12,5 g/l	1.1	5	5	15	15	20
T=55, C=12,5 g/l	2.1	5	5	15	20	15
T=55, C=12,5 g/l	3.1	5	10	10	20	20
T=65, C=12,5g/l	6	15	20	20	25	25
T=65, C=12,5g/l	3	20	10	35	40	40

**Multidraw KTL N16**

T=50, C=10g/l	3	65	65	65	80	80
T=50, C=10g/l	1	60	50	50	55	55
T=55, C=10g/l	5	55	80	90	90	
T=55, C=10g/l	4	65	80	95		
T=60, C=10g/l	4	85	80	85	90	
T=60, C=10g/l	1	30	30	35	50	70
T=65, C=10g/l	2	75	65	80	80	90
T=65, C=10g/l	5	80	80	90	95	95
T=50, C=12,5g/l	1	50	50	60	70	80
T=50, C=12,5g/l	2	40	60	65	65	85
T=55, C=12,5 g/l	1.1	50	50	60	70	80
T=55, C=12,5 g/l	2.1	40	60	65	65	85
T=55, C=12,5 g/l	3.1	45	50	60	75	90
T=65, C=12,5g/l	2	90	90	95		
T=65, C=12,5g/l	3	90	90	95		

**SW4020/HDG**

T=50, C=10g/l	1	5	5	5	10	10
T=50, C=10g/l	3	5	5	5		
T=55, C=10g/l	2	5	5	5		
T=55, C=10g/l	3	5	5	5		

**Anticorit RP 4107S/HDG**

T=50, C=10g/l	1	60	80	80	85	85
T=50, C=10g/l	3	40	60	60	70	80
T=55, C=10g/l	2	70	70	70	70	70
T=55, C=10g/l	3	60	80	80	80	80
T=60, C=10g/l	2	85	85	90	90	

**Binol Cut 10/HDG**

T=50, C=10g/l	1	5	5	10	15	15
T=50, C=10g/l	3	5	5	5	10	10
T=55, C=10g/l	1	5	5	5		
T=60, C=10g/l	2	5	5	5		

**Binol Cut 20/GA**

T=50, C=10g/l	1	15	20	30	30	30
T=50, C=10g/l	3	10	20	25	30	35
T=55, C=10g/l	6	15	20	20	50	60
T=55, C=10g/l	5	20	20	40	65	80
T=60, C=10g/l	3	10	20	90	80	85

T=60, C=10g/l	2	25	50	60	75	90
T=65, C=10g/l	2	20	60	75	80	90
T=65, C=10g/l	5	10	25	70	80	85
T=50, C=12,5g/l	6	20	25	60	85	85
T=50, C=12,5g/l	4	20	30	55	80	80
T=65, C=12,5g/l	4	15	40	85	90	95
T=65, C=12,5g/l	1	20	50	80	90	
<b>SW4020/EG</b>						
T=50, C=10g/l	5	5	5	5	5	5
T=50, C=10g/l	6	5	5	10		
T=55, C=10g/l	2	5	5	5		
T=55, C=10g/l	3	5	5	5		
T=60, C=10g/l	4	5	5	5		
T=60, C=10g/l	1	5	10	10		
T=65, C=10g/l	2	5	5	10		
T=65, C=10g/l	5	5	5	5		
T=50, C=12,5g/l	1	5	5	10		
T=50, C=12,5g/l	3	5	5	10	10	
T=65, C=12,5g/l	4	5	5	10	15	25
T=65, C=12,5g/l	6	5	10	10		
<b>SW4020/CRS</b>						
T=50, C=10g/l	2	5	5	10		
T=50, C=10g/l	1	5	5	10	30	40
T=55, C=10g/l	6	10	30	60	75	75
T=55, C=10g/l	4	5	5	10	45	60
T=60, C=10g/l	5	10	20	60	80	95
T=60, C=10g/l	1	5	10	10	55	95
T=65, C=10g/l	3	10	20	60	70	95
T=65, C=10g/l	6	15	25	75	100	
T=50, C=12,5g/l	3	5	5	55	65	80
T=50, C=12,5g/l	4	10	20	35	60	80
T=65, C=12,5g/l	5	30	70	85	90	
T=65, C=12,5g/l	2	10	10	25	40	65
<b>Umformöl 60/HDG</b>						
T=50, C=10g/l	1	5	10	10	10	10
T=50, C=10g/l	2	5	5	10		
T=55, C=10g/l	3	5	5	10	10	10
T=55, C=10g/l	5	5	5	10	10	10
T=60, C=10g/l	4	5	10	5	10	15
T=60, C=10g/l	6	10	10	10	5	10
T=65, C=10g/l	5	5	10	10		
T=65, C=10g/l	3	5	10	10		
T=50, C=12,5g/l	4	5	10	5	5	
T=50, C=12,5g/l	6	5	5	10		
T=65, C=12,5g/l	2	5	5	5		
T=65, C=12,5g/l	1	20	10	10	10	15

**Table 4.** Result from the test method by increasing the parameters.

**Draw 362/HDG** gave very good results. All the oil has been degreased from the substrate, even though the amount of oil is high. *See appendix 1.*

**Aral Ropa W35/HDG:** Covered in a very high amount of oil on the substrate, but still not effecting the result badly. Aral Ropa W35 is emulsions oil, so this makes it easier to degrease the surface. The other emulsion oils tested in the first test, read report ER-513386, all received a very good result therefore no more test will be run.

**Iloform PL6/HDG:** Received a not a very good result in last experiment. When the parameters were increased a slight better results were achieved at a higher temperature.

**Aral Ropa 4093/HDG:** Obtained a bad result so the parameters were increased, and the percentages increased around fifteen percent.

**Multidraw KTL N16/HDG:** In this experiment it achieved a very good result. Even at a lower temperature and a lower concentration, and also when the parameters were increased. At three panels there were very high amount of oil; still it did not effect the result. *See appendix 1.*

**SW4020/HDG:** Very poor percentage result was given at tested parameters. No further test was made, since the oil when used in the production will be precured.

**Anticorit RP 4107S/HDG:** Received a very good result in this experiment. A better result may be achieved if a strictly maximum of 2 g/m<sup>2</sup> is hold. *See appendix 1.*

**Binol Cut 10/HDG:** Very poor percentage result was given at tested parameters. No further test was made, since the oil when used in the production will be precured.

**Binol Cut 20/GA:** Increased in percentage from a result that was not a very good one to a result that was very good when the temperature was increased

**SW4020/EG:** From this test a very poor result was received even when the parameters were varied.

**SW4020/CRS:** Increased in percentage from a result that was not a very good one to a result that was very good when the temperature was increased. Panel two in the test however had a higher amount of oil then the other panels, maybe therefore gave the minor percent of water film.

**Umformöl 60/HDG:** Very poor result was given. No improvement when increases the parameters.

### 5.3.1 Test with maximum temperature and concentration

Three of the oils that were tested in the previous test gave very poor result. One more test has been running but this time the temperature were; 65 degrees and the concentration 15 g/l. Same procedure is repeated as before with preparation and with the test method.

Oil/Substrate	Nr.	Cycle 1	2	3	4	5
<b>Aral Ropa 4093/HDG</b>						
T=65, C=15g/l	1.1.	15	10	15	25	25
T=65, C=15g/l	1.2	10	10	10	15	30
<b>SW4020/EG</b>						
T=65, C=15g/l	1.1.	5	5	20	50	95
T=65, C=15g/l	1.2	5	5	25	60	95
<b>Umformöl 60/HDG</b>						
T=65, C=15g/l	1.1	5	5	5	5	10
T=65, C=15g/l	1.2	5	5	5	10	10

*Table 5.* Data table with result from the last and third batch with a maximum temperature and concentration.

A successful result was reached with SW4020/EG. All the oil was almost defatted at the higher temperature and with the higher concentration

No better results were achieved for Aral Ropa 4093/HDG with higher temperature and higher concentration and the same is for the oil Umformöl 60/HDG.

Temperature and concentration has not been increased further, since the laboratory method should not deviate too much from the parameters used in the production process.

### 5.3.2 Result from other methods

#### Contact Angle

The results have been received from repeated test at least two times and the table show means values of the diameter and the contact angle of the oils. The diameters of the drops are measured in millimeters and the contact angles are read in table and are measured in degrees.

Oil/Substrate	Diameters (mm)	Contact Angle (degrees)
Draw 362/HDG	6,0	26,1
Aral Ropa W35/HDG	8,2	-
Iloform PL6/HDG	7,0	16,8
Aral Ropa 4093/HDG	8,0	11,3
Multidraw KTL N16/HDG	4,8	46,9
Anticorit RP 4107S/HDG	4,0	69,0
Umformöl 60/HDG	3,7	78,8
SW4020/EG	6,8	18,2
SW4020/CRS	6,9	17,5
Binol Cut 10/HDG	7,5	13,7
Binol Cut 20/GA	7,8	12,2

*Table 6.* Result of calculating the contact angles of the oils.

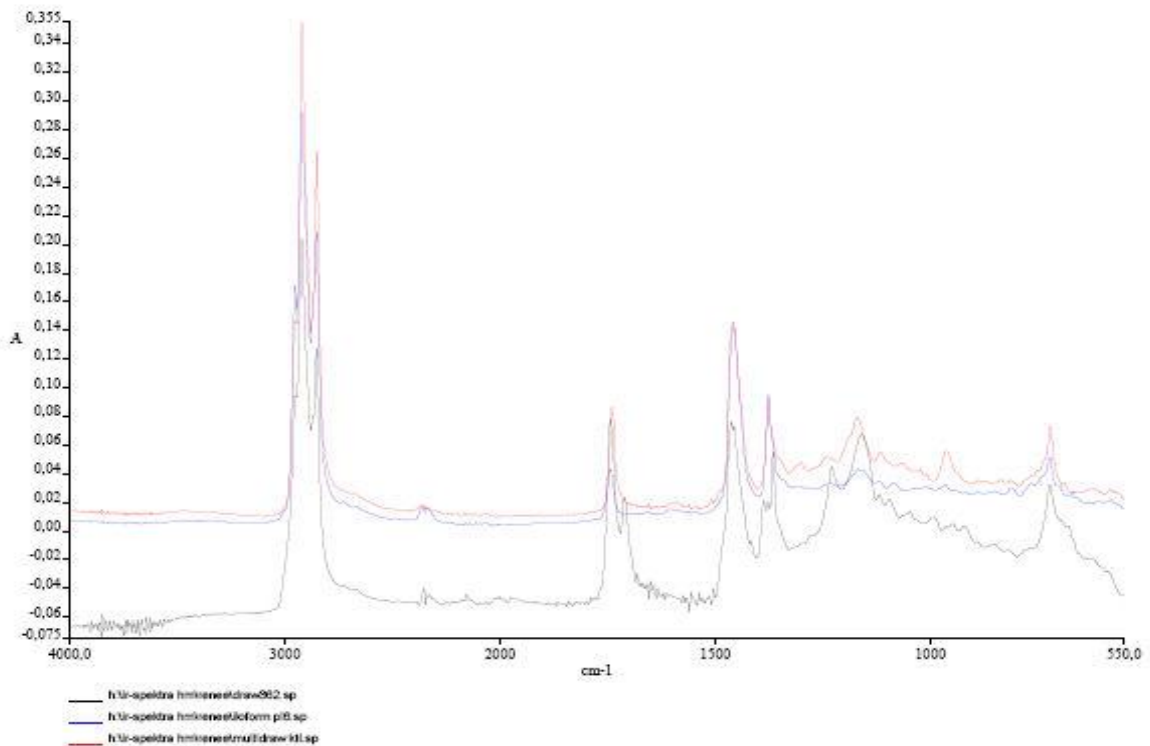
#### IR

Mineral oils show peaks at wavelengths 2920, 1462, 1365 and 722 due to absorption by carbon hydrogen bonds. This can be seen in all three spectra so it is evident that mineral oil is

a substantial component of all oils. The peaks at 1740 and 1160 are from fatty acid ester showing that this component also is present in all of the three oils.

This show that these three oils Draw 362, Iloform PL6 and Multidraw KTL N16 are closely related but there is still a substantial difference to the different spectra. In the spectra can be seen that Draw 362 has a higher peak of fatty acid ester than the other oils contain.

To be able to distinguish exactly the substances and hold more information another tool are needed than IR for example GC-MS. [16]



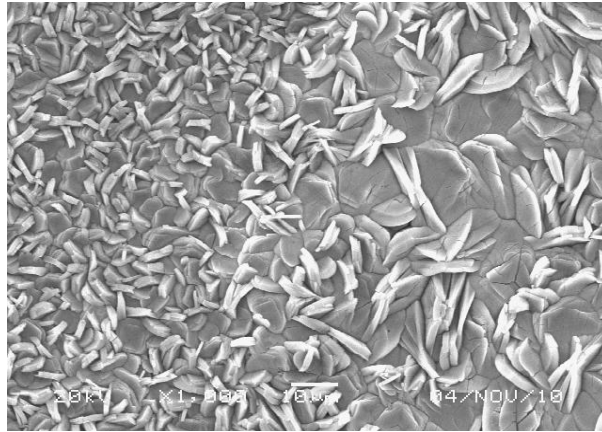
**Fig. 3:** IR- spectra for all the three oils – Draw 362, Iloform PL6 and Multidraw KTL N16. [16] See appendix 5.

## SEM

The panels that looked visually spotted from the production still meets the requirements. However, at certain areas of the substrate the crystals are lying down and some of the crystals are larger than guidelines say, which in turn can lead to a bad adhesion.

Four of the twelve oils that were analyzed using SEM had observations. They had a little tendency to larger crystals and some areas where the crystals were lying down.

Multidraw KTL N16 for example was run through the production twice at two different occasions and still showed a good result in the SEM, even though the substrates had visible spots on the surface from production at both times. See more pictures in Appendix 6.



**Fig 4.** Analyzed with SEM, SW4020/HDG nr.2, magnification x1000. A normal area with fine crystals is shown on left hand side of the picture, which is next to an area where the crystals are larger than 10 $\mu$ m and the crystals are lying down.

## 6 Conclusions

- From the experiment, temperature 50 degrees and concentration 10 g/l, comparing the results from the report ER- 513386 achieved almost the same results. Some of them gave opposite results. Reasons may be that the amount of oil is different in the two experiments, the preparation and execution of the method may differ.
- Some of the oils showed very poor results at a temperature of 50 degrees and a concentration of 10 g/l. When the parameters were increased, few of them gave a better result at a higher temperature and also with a higher concentration. In most of the cases, it does not matter if both temperature and concentration is increased to obtain a better result. Thus, since the degreasing agent Gardoclean 855/1 is highly alkaline and corrosive, it is better not to try to increase the concentration if it is not really needed. Therefore keep the concentration of 10 g/l and raise the temperature.
- The oils that already had a good percentage of water film improved even further with increasing the concentration and the temperature.
- Higher temperature in the water bath is suggested, which might improve the results since there is such a large temperature difference between the two beakers.
- It has shown that some oils after rinsing in the water bath seems to be difficult to degrease in the following cycle. Perhaps it is better to begin dipping into the deionized water beaker after the test panel has been dipped three times each for 30 seconds into the degreasing bath that is the first two cycles without the rinsing step.
- Another reflection is whether it is necessary to stir the panel for 30 seconds, or if there is only enough to keep the panel still in the degreasing bath for 30 seconds. The time is one parameter that can be look at and see if it is possible to shorten it or if prolonging the time can give improved results.
- The method was also compared to production. Test panels went through the pre-treatment process followed by a visual assessment of the phosphate layer. Most panels were good but some of them had spots on their surface. Investigation by SEM only showed minor disturbances. However, the results from the laboratory method were not always consistent with the results from the production process.
- Some of the phosphated panels that were visually spotted from production showed nothing unusual in the SEM analysis. A visual assessment is no guarantee that it will be a bad result in laboratory or in production.

An analysis could be recommended also with the substrate after degreasing in the laboratory, to see if something is left of on the substrate.

- What happens to the oil if the oil is not totally degreased from the three steps from the production? Does the phosphating step remove the remaining oil on the substrate so therefore the crystals are not damaged nor have any remarks? This should be

investigated further what is happening with the layer of oil which is not removed completely from the degreasing steps.

- The mechanical cleaning by high pressure and spray degreasing in production are important steps, which is a great difference compared to the degreasing in laboratory with only cleaning by dipping and therefore it may be a reason to the difference in the results from laboratory compared to the production.
- Binol Cut 10 is a vegetable oil; there may be a link to the remarks from the production. Since the content from the data sheets can not provide any context, it is difficult to state this claim. In further experiment, this should be considered.
- For example, SW4020/CRS created more water film at a higher temperature. One of the panels that were tested had a higher amount of oil than the other test panels and this panel did not give the same good results as the others even when tested at 65 degrees and with a concentration of 12, 5 g/l. Thus, the amount of oil, in this case should be around 1, 5 g/m<sup>2</sup> instead.
- In generally, it seems best to keep down the amount of oil, where a maximum should be at 2, 0 g/m<sup>2</sup>. But for some of the oils, it made no difference whether the amount of oil were higher than what is recommended.
- SW4020/EG went from a bad percentage result to a very good result when the parameters were increased to its maximum. The other two oils, which also were tested, did not improve results. It does not seem to be an idea in general to have such a high concentration with such a high temperature.
- All content of compounds in the oils is not declared in the data sheet. With this vague information, no connection could be drawn between all the oils. It is recommended to contact the suppliers for more information of the oils; the VCC has the right to get more information even when the information is confidential.
- To be able to distinguish exactly the substances and get more information about the oils requires other tools and method than IR.
- Calculating the contact angle did not give correlations between the oils. Found no link such as a rule that more viscous oils had a lower contact angle than them less viscous. When there is an uncertainty regarding the method, the method should not be rejected in upcoming trial.
- It is not possible to say that the method is reliable when it does not provide a concrete result of all oils tested. Overall it was a better performance when parameters were changed with some exceptions. Individual oil should be considered and evaluated by their own characteristics and not only based on standard methodology, but instead try to relate to this experiment how it can be performed.

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## Appendix 1: Data results from laboratory

Prepared	Nr.	Oil/ Substrate	Mix (%)	Weight before oil (g)	weight after oil (g)	weight oil (g)	Area (m2)	Amount of oil (g/m2)
2010-09-06	1	Draw 362/HDG	100	125,6027	125,977	0,3743	0,04	9,36
2010-09-06	2	Draw 362/HDG	100	125,7381	126,128	0,3899	0,04	9,74
2010-09-06	3	Draw 362/HDG	100	126,0457	126,4332	0,3875	0,04	9,69
2010-09-06	1	Aral Ropa W35/HDG	100	125,6088	126,7991	1,1903	0,04	29,76
2010-09-06	2	Aral Ropa W35/HDG	100	126,5062	127,7892	1,283	0,04	32,10
2010-09-06	3	Aral Ropa W35/HDG	100	126,8266	128,026	1,1994	0,04	30,00
2010-09-06	1.1	Iloform PL6/HDG	100	126,9932	127,0401	0,0469	0,04	1,176
2010-09-06	2.1	Iloform PL6/HDG	100	125,8971	125,9442	0,0471	0,04	1,18
2010-09-06	3.1	Iloform PL6/HDG	100	126,6824	126,7323	0,0499	0,04	1,24
2010-09-09	1.2	Iloform PL6/HDG	100	125,4834	125,5496	0,0662	0,04	1,66
2010-09-09	2.2	Iloform PL6/HDG	100	127,1179	127,1847	0,0668	0,04	1,72
2010-09-09	3.2	Iloform PL6/HDG	100	127,0297	127,0982	0,0685	0,04	1,71
2010-09-15	1	Iloform PL6/HDG	100	127,0526	127,0986	0,04	0,04	1,15
2010-09-15	2	Iloform PL6/HDG	100	127,0227	127,0702	0,0475	0,04	1,19
2010-09-15	3	Iloform PL6/HDG	100	126,9117	126,956	0,0443	0,04	1,11
2010-09-15	4	Iloform PL6/HDG	100	126,662	126,71	0,048	0,04	1,20
2010-09-15	5	Iloform PL6/HDG	100	125,2489	125,2931	0,0442	0,04	1,10
2010-09-15	6	Iloform PL6/HDG	100	126,7396	126,7848	0,0452	0,04	1,13
2010-09-09	1.1	Aral Ropa 4093/HDG	100	126,8544	126,9842	0,1298	0,04	3,24
2010-09-09	2.1	Aral Ropa 4093/HDG	100	125,3392	125,4523	0,1131	0,04	2,82
2010-09-09	3.1	Aral Ropa 4093/HDG	100	125,1988	125,3152	0,1164	0,04	2,91
2010-09-15	1	Aral Ropa 4093/HDG	100	127,0428	127,1251	0,0823	0,04	2,06
2010-09-15	2	Aral Ropa 4093/HDG	100	125,8589	125,9393	0,0804	0,04	2,01
2010-09-15	3	Aral Ropa 4093/HDG	100	126,9769	127,0613	0,0844	0,04	2,11
2010-09-15	4	Aral Ropa 4093/HDG	100	126,9309	127,0114	0,0805	0,04	2,01
2010-09-15	5	Aral Ropa 4093/HDG	100	125,6963	125,7755	0,0792	0,04	1,98
2010-09-15	6	Aral Ropa 4093/HDG	100	126,3032	126,381	0,0778	0,04	1,94
2010-09-09	1.1	MultidrawKTLN16/HDG	100	126,2241	126,5573	0,3332	0,04	8,33
2010-09-09	2.1	MultidrawKTLN16/HDG	100	125,9998	126,3339	0,3341	0,04	8,36
2010-09-09	3.1	MultidrawKTLN16/HDG	100	126,2241	126,5573	0,3332	0,04	8,33
2010-09-27	1	MultidrawKTLN16/HDG	100	125,5343	125,6189	0,0846	0,04	2,12
2010-09-27	2	MultidrawKTLN16/HDG	100	127,1938	127,3126	0,1188	0,04	2,97
2010-09-27	3	MultidrawKTLN16/HDG	100	126,5432	126,69	0,1468	0,04	3,67
2010-09-27	4	MultidrawKTLN16/HDG	100	126,9988	127,0924	0,0936	0,04	2,34
2010-09-27	5	MultidrawKTLN16/HDG	100	126,4934	126,5851	0,0917	0,04	2,29
2010-09-22	1	SW4020/HDG	100	125,531	125,5939	0,0629	0,04	1,57
2010-09-22	2	SW4020/HDG	100	125,1144	125,1855	0,0711	0,04	1,78
2010-09-22	3	SW4020/HDG	100	126,6191	126,6957	0,0766	0,04	1,92
2010-09-22	1	AnticoritRP4107S/HDG	100	126,3459	126,4576	0,1117	0,04	2,79
2010-09-22	2	AnticoritRP4107S/HDG	100	125,7529	125,8739	0,121	0,04	3,03
2010-09-22	3	AnticoritRP4107S/HDG	100	126,0448	126,1762	0,1314	0,04	3,29

2010-09-22	1	Binol Cut 10/HDG	100	125,9338	126,0124	0,0786	0,04	1,97
2010-09-22	2	Binol Cut 10/HDG	100	127,1436	127,2152	0,0716	0,04	1,79
2010-09-22	3	Binol Cut 10/HDG	100	126,2378	126,3094	0,0716	0,04	1,79
2010-09-27	1	Binol Cut 20/GA	100	102,5984	102,7103	0,1119	0,04	2,80
2010-09-27	2	Binol Cut 20/GA	100	102,5	102,6056	0,1056	0,04	2,64
2010-09-27	3	Binol Cut 20/GA	100	103,9878	104,0984	0,1106	0,04	2,77
2010-09-27	4	Binol Cut 20/GA	100	103,7846	103,8933	0,1087	0,04	2,72
2010-09-27	5	Binol Cut 20/GA	100	104,742	104,8531	0,1111	0,04	2,78
2010-09-27	6	Binol Cut 20/GA	100	104,3627	104,471	0,1083	0,04	2,71
2010-09-29	1	SW4020/EG	100	116,3476	116,4296	0,082	0,04	2,05
2010-09-29	2	SW4020/EG	100	116,2488	116,3292	0,0804	0,04	2,01
2010-09-29	3	SW4020/EG	100	116,6533	116,7343	0,081	0,04	2,02
2010-09-29	4	SW4020/EG	100	117,9845	118,0709	0,0864	0,04	2,16
2010-09-29	5	SW4020/EG	100	117,822	117,9107	0,0887	0,04	2,22
2010-09-29	6	SW4020/EG	100	115,2363	115,3147	0,0784	0,04	1,96
2010-10-05	1	SW4020/CRS	100	121,3766	121,4322	0,0556	0,04	1,39
2010-10-05	2	SW4020/CRS	100	120,9456	121,0384	0,0928	0,04	2,32
2010-10-05	3	SW4020/CRS	100	120,7543	120,8155	0,0612	0,04	1,53
2010-10-05	4	SW4020/CRS	100	120,7386	120,8045	0,0659	0,04	1,65
2010-10-05	5	SW4020/CRS	100	121,7144	121,7789	0,0645	0,04	1,61
2010-10-05	6	SW4020/CRS	100	121,6621	121,7229	0,0608	0,04	1,52
2010-10-05	1	Umformöl 60/HDG	100	126,4056	126,4774	0,0718	0,04	1,80
2010-10-05	2	Umformöl 60/HDG	100	124,9443	125,0088	0,0645	0,04	1,61
2010-10-05	3	Umformöl 60/HDG	100	127,2186	127,289	0,0704	0,04	1,76
2010-10-05	4	Umformöl 60/HDG	100	126,3438	126,4134	0,0696	0,04	1,74
2010-10-05	5	Umformöl 60/HDG	100	126,8259	126,8939	0,068	0,04	1,70
2010-10-05	6	Umformöl 60/HDG	100	127,0648	127,1299	0,0651	0,04	1,63

**Fig. A.** Data sheets for measuring the panels and calculating the amount of oil.

Prepared	Nr.	Oil/ Substrate	Mix (%)	Weight before oil (g)	weight after oil (g)	weight oil (g)	Area (m2)	Amount of oil (g/m2)
2010-10-25	1	Aral Ropa4093/HDG	100	126,2618	126,3573	0,0955	0,04	2,39
2010-10-25	1	SW4020/EG	100	116,2709	116,3551	0,0842	0,04	2,10
2010-10-25	1	Umformöl 60/HDG	100	126,1946	126,2897	0,0951	0,04	2,38

**Fig. B.** Data sheets for measuring the panels and calculating the amount of oil

## Appendix 2: Data result from production

Prepared	Oil/ Substrate	Nr	Mix (%)	Weight before oil (g)	weight after oil (g)	weight oil (g)	Area (m2)	Amount of oil (g/m2)
2010-09-06	Draw 362/HDG	1	100	126,6446	127,0389	0,3943	0,0399	9,87
2010-09-06	Draw 362/HDG	2	100	126,4493	126,8329	0,3836	0,0399	9,60
2010-09-06	Draw 362/HDG	3	100	127,1502	127,5403	0,3901	0,0399	9,77
2010-09-06	Aral Ropa W35/HDG	1	100	126,7216	127,964	1,2424	0,0399	31,06
2010-09-06	Aral Ropa W35/HDG	2	100	126,8498	128,1001	1,2503	0,0399	31,26
2010-09-06	Aral Ropa W35/HDG	3	100	126,579	127,7835	1,2045	0,0399	30,11
2010-09-06	Iloform PL6/HDG	1.1	100	126,4821	126,5288	0,0467	0,0399	1,17
2010-09-06	Iloform PL6/HDG	2.1	100	126,1845	126,2281	0,0436	0,0399	1,09
2010-09-06	Iloform PL6/HDG	3.1	100	125,3984	125,4418	0,0434	0,0399	1,09
2010-09-09	Iloform PL6/HDG	1.2	100	126,691	126,7666	0,0756	0,04	1,89
2010-09-09	Iloform PL6/HDG	2.2	100	126,6132	126,6915	0,0783	0,04	1,96
2010-09-09	Iloform PL6/HDG	3.2	100	126,0109	126,0866	0,0757	0,04	1,90
2010-09-09	Aral Ropa 4093/HDG	1	100	125,5858	125,7052	0,1167	0,0399	2,99
2010-09-09	Aral Ropa 4093/HDG	2	100	126,1217	126,2464	0,1247	0,0399	3,12
2010-09-09	Aral Ropa 4093/HDG	3	100	125,7101	125,8352	0,1251	0,0399	3,13
2010-09-09	Multidraw KTL N16/HDG	1.1	100	126,8555	127,1719	0,3164	0,0399	7,92
2010-09-09	Multidraw KTL N16/HDG	2.1	100	126,5272	126,8654	0,3382	0,0399	8,47
2010-09-09	Multidraw KTL N16/HDG	3.1	100	126,4787	126,7799	0,3012	0,0399	7,54
2010-09-27	Multidraw KTL N16/HDG	1.2	100	126,5044	126,598	0,0936	0,0399	2,34
2010-09-27	Multidraw KTL N16/HDG	2.2	100	126,9298	127,0163	0,0865	0,0399	2,17
2010-09-27	Multidraw KTL N16/HDG	3.2	100	126,5096	126,6527	0,1431	0,0399	3,58
2010-09-22	SW4020/HDG	1	100	125,7614	125,8426	0,0812	0,0399	2,03
2010-09-22	SW4020/HDG	2	100	125,2873	125,3621	0,0748	0,0399	1,87
2010-09-22	SW4020/HDG	3	100	126,2171	126,3049	0,0878	0,0399	2,20
2010-09-22	Anticorit RP 4107S/HDG	1	100	126,3422	126,4539	0,1117	0,0399	2,80
2010-09-22	Anticorit RP 4107S/HDG	2	100	125,3574	125,484	0,1266	0,0399	3,17
2010-09-22	Anticorit RP 4107S/HDG	3	100	125,8891	126,0008	0,1117	0,0399	2,80
2010-09-22	Binol Cut 10/HDG	1	100	126,6163	126,6787	0,0624	0,0399	1,56
2010-09-22	Binol Cut 10/HDG	2	100	126,7153	126,7768	0,0615	0,0399	1,54
2010-09-22	Binol Cut 10/HDG	3	100	126,7459	126,8104	0,0645	0,0399	1,61
2010-09-27	Binol Cut 20/GA	1	100	104,4209	104,5293	0,1084	0,0399	2,71
2010-09-27	Binol Cut 20/GA	2	100	103,9677	104,0681	0,1004	0,0399	2,51
2010-09-27	Binol Cut 20/GA	3	100	103,9595	104,0582	0,0987	0,0399	2,47
2010-09-29	SW4020/EG	1	100	117,3206	117,4	0,0794	0,0399	1,99
2010-09-29	SW4020/EG	2	100	116,5577	116,6382	0,0805	0,0399	2,02
2010-09-29	SW4020/EG	3	100	115,2572	115,3588	0,1016	0,0399	2,54
2010-10-05	SW4020/CRS	1	100	120,9116	120,9775	0,0659	0,0399	1,65
2010-10-05	SW4020/CRS	2	100	121,2614	121,3233	0,0619	0,0399	1,55
2010-10-05	SW4020/CRS	3	100	120,1214	120,1874	0,066	0,0399	1,65
2010-10-05	Umformöl 60/HDG	1	100	126,278	126,3462	0,0682	0,0399	1,71
2010-10-05	Umformöl 60/HDG	2	100	125,2468	125,3015	0,0547	0,0399	1,37
2010-10-05	Umformöl 60/HDG	3	100	125,9945	126,0638	0,0693	0,0399	1,73

Fig. C. Data sheets for measuring the panels and calculating the amount of oil.

### Appendix 3: Specimens with no sign of defects

The stripes on the panels are from the production and are not caused by the degreasing test.



**Fig. D.** SW2040/HDG on panel number one, Binol Cut 10 on panel number one and Anticorit RP4107S on panel number three.

## Appendix 4: Specimens with defects

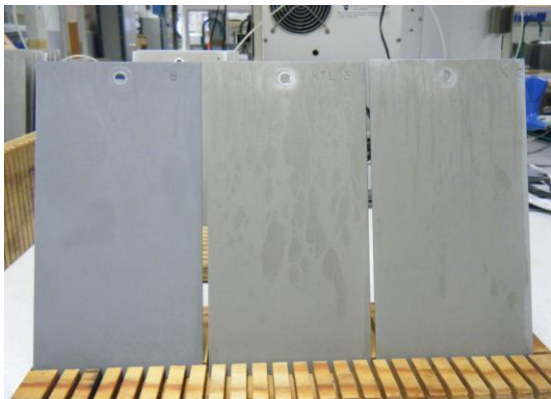
The stripes on all the panels are from the production and are not caused by the degreasing test.



**Fig. E.** Overview of panels Multidraw KTL N16/HDG and Anticorit RP 4107S/HDG.



**Fig. F.** Umförmol 60/HDG contains spots and white dots on the substrate on panel number one and two. These white dots is due from the production.



**Fig. G.** Binol Cut 20/GA panel number one, Multidraw KTL N16/HDG panel number two from batch one and Multidraw KTL N16/HDG panel number two in batch two have spots on them.

## Appendix 5: IR

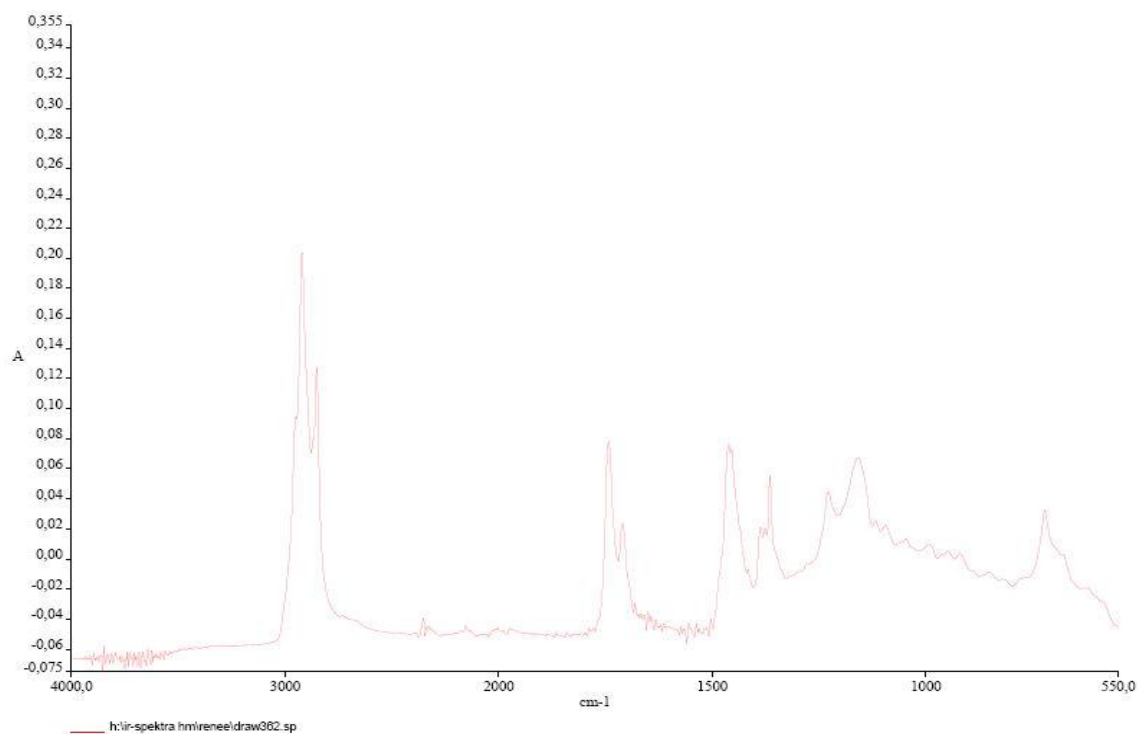


Fig H: IR-spectra for Draw 362 [16]

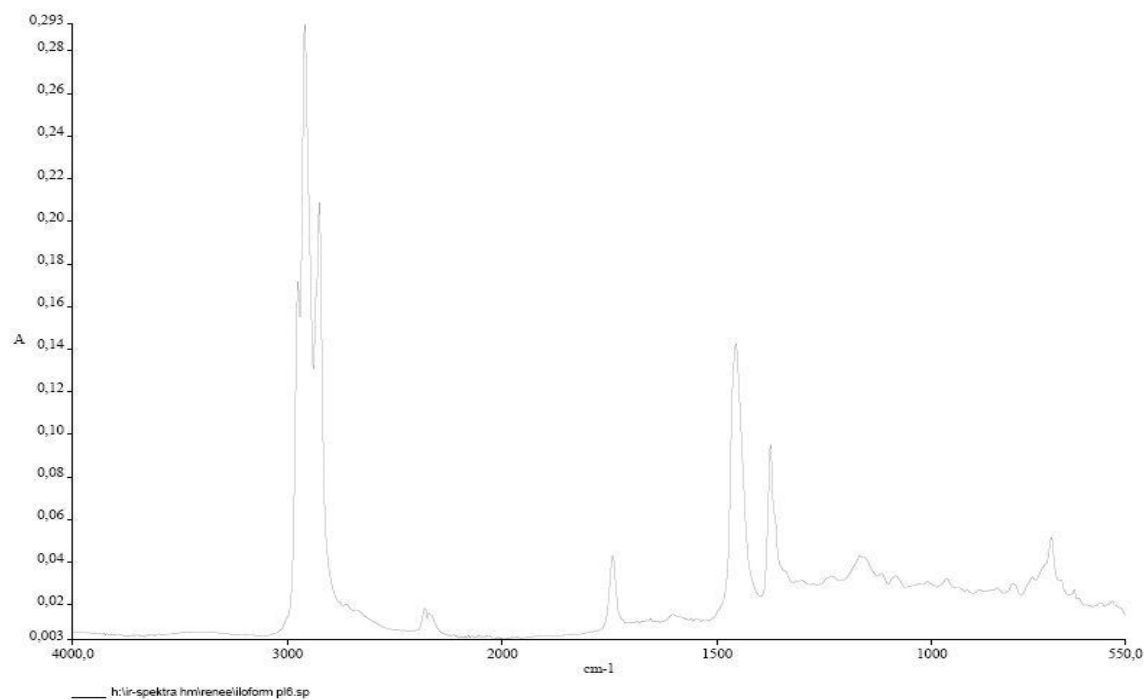
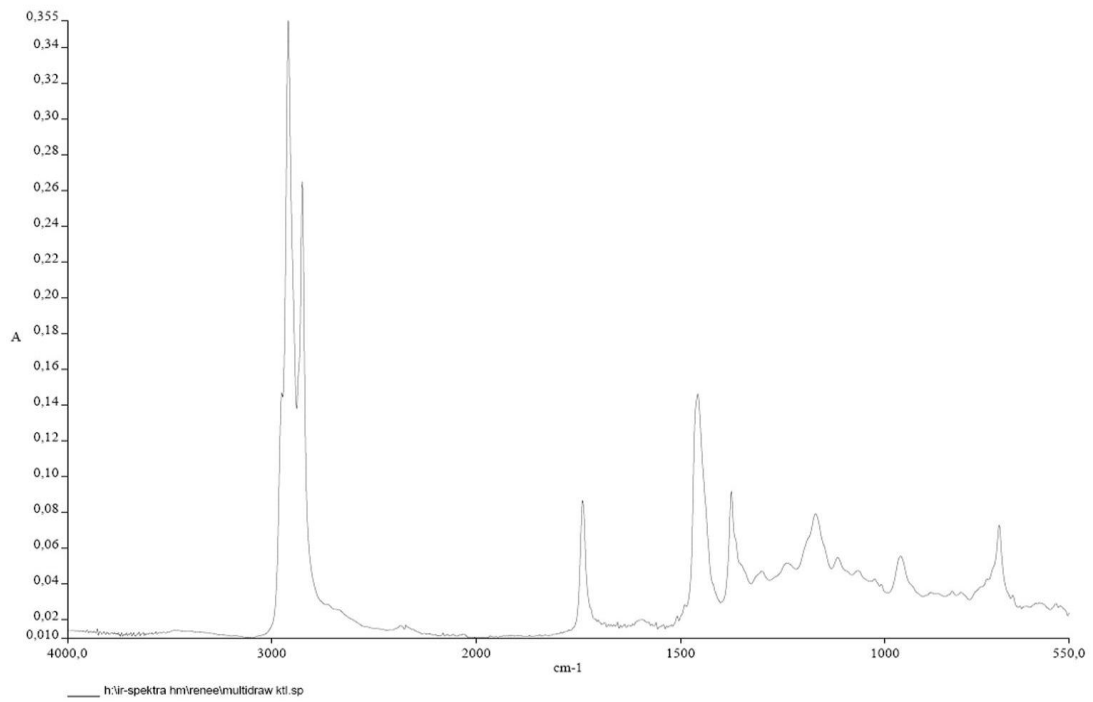
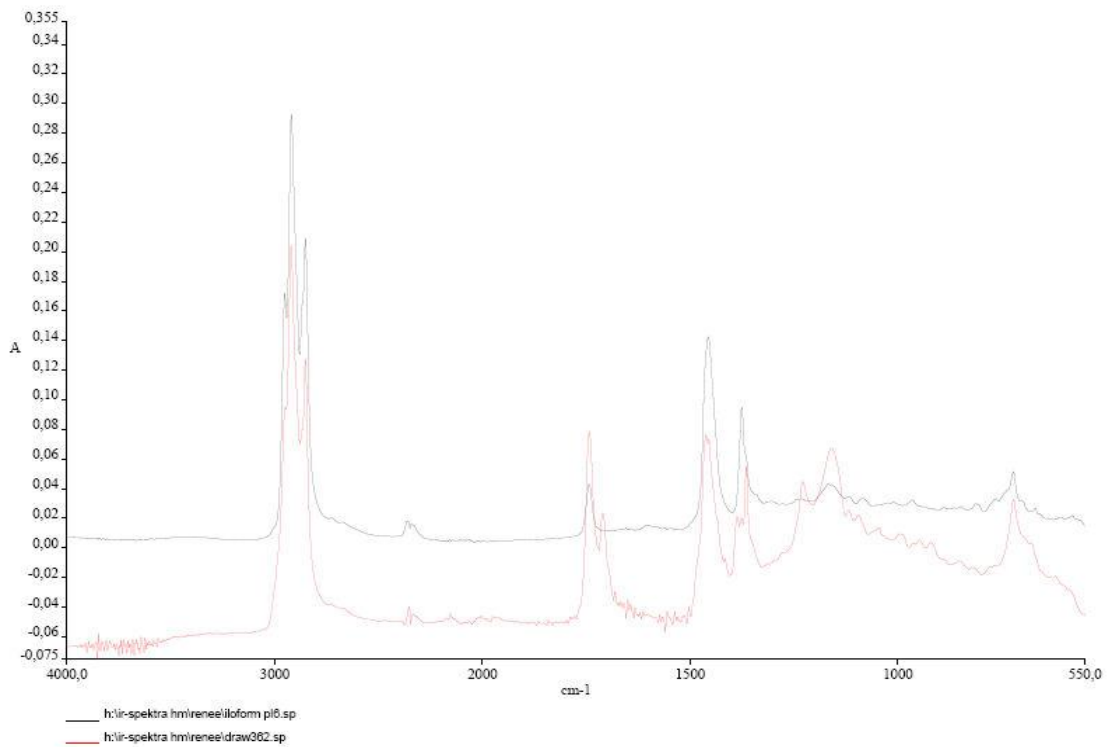


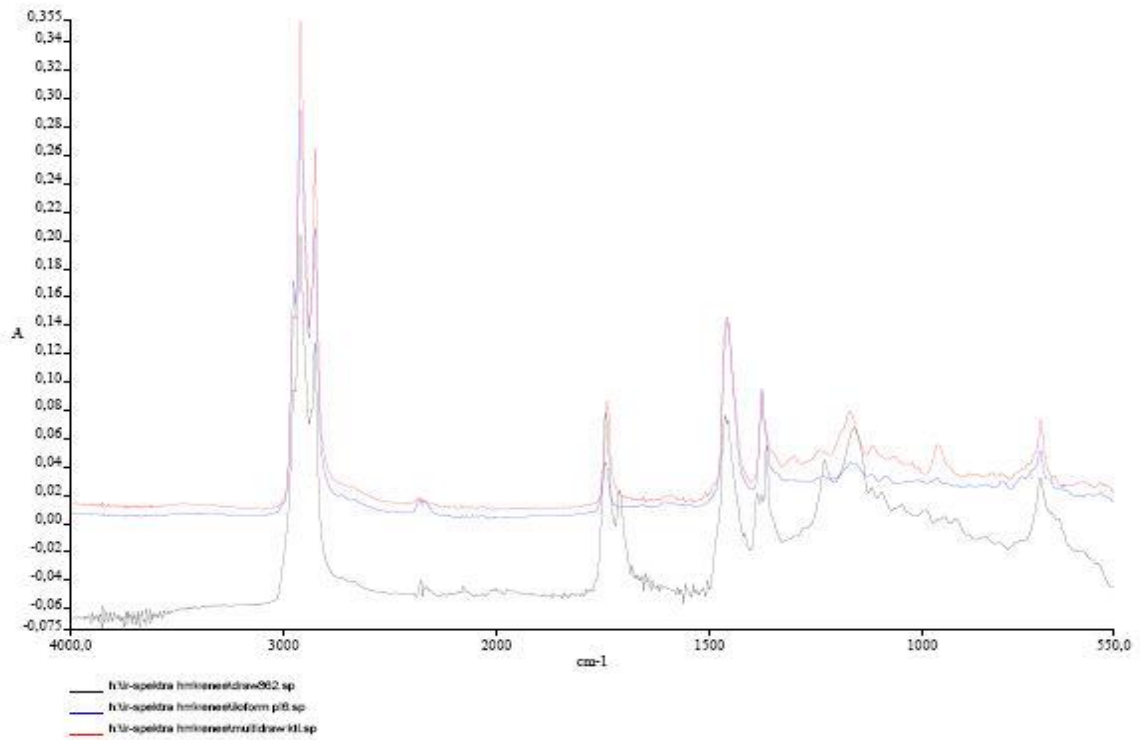
Fig. I: IR-spectra for Iloform PL6 [16]



**Fig. J:** IR-spectra for Multidraw KTL N16 [16]



**Fig. K:** IR- spectra with Iloform PL6 and Draw 362 [16]



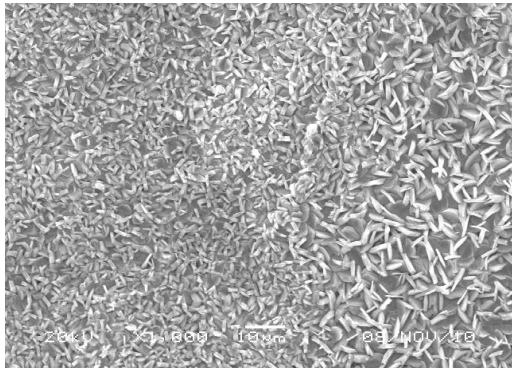
**Fig. L:** IR- spectra for all the three oils – Draw 362, Iloform PL6 and Multidraw KTL N16. [16]

## Appendix 6: SEM

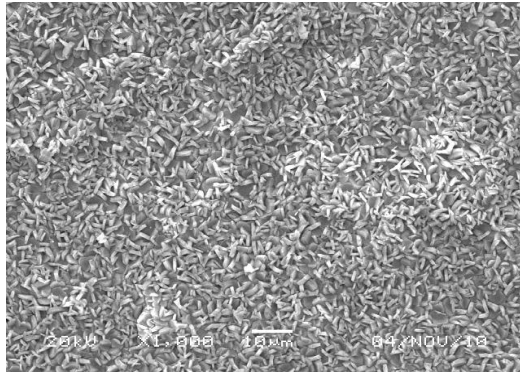
Normal area - visually without any remarks on the panel's surface.

Spotted area – visually spotted on the panels surface

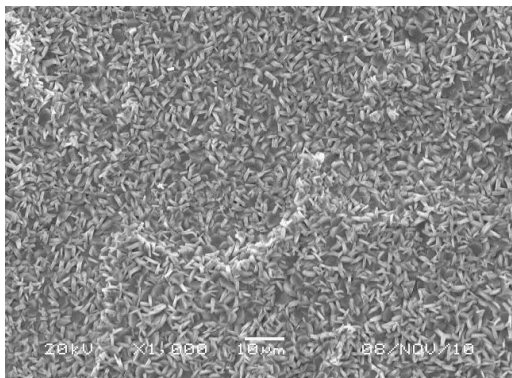
### Round 1: Went through the production 2010-09-07



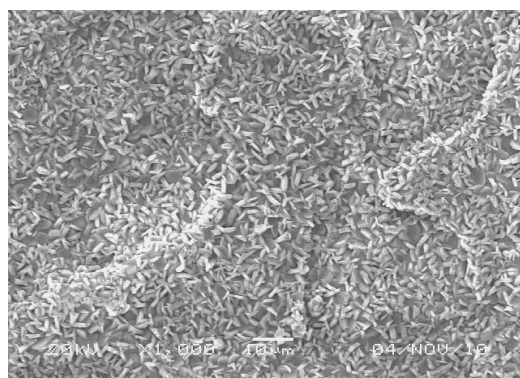
Iloform PL6/HDG plate nr.2,  
normal area, x1000.



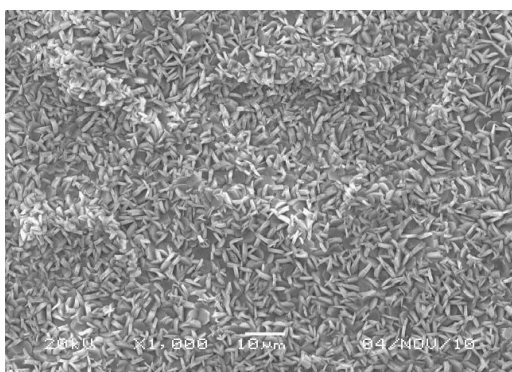
Iloform PL6/HDG nr.1,  
normal area, x1000



Aral Ropa W35/HDG nr.3,  
normal area, x1000

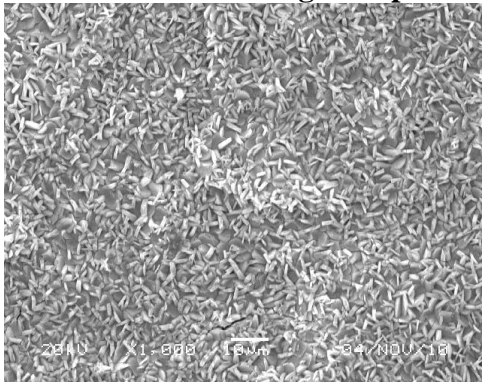


Aral Ropa W35/HDG nr.2,  
normal area, x1000

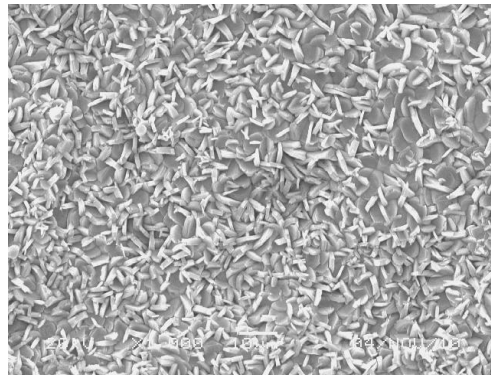


Draw 362/HDG nr.1,  
normal area, x1000

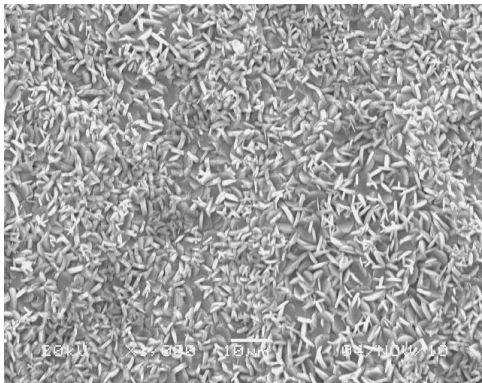
**Round 2: Went through the production 2010-09-10**



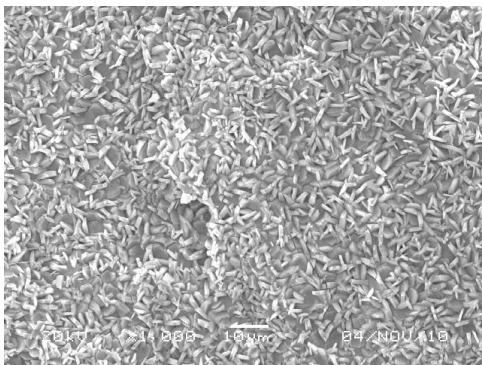
Aral Ropa 4093/HDG nr.1,  
normal and a spotted area, x1000



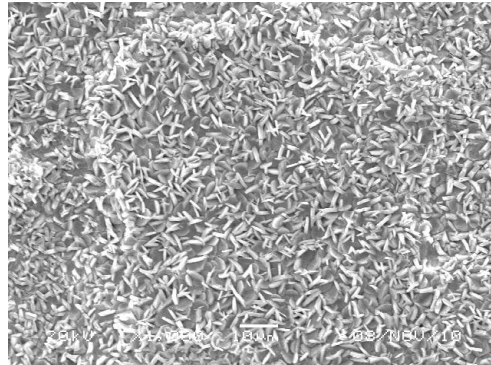
Aral Ropa 4093/HDG nr. 3,  
normal and spotted area, x1000



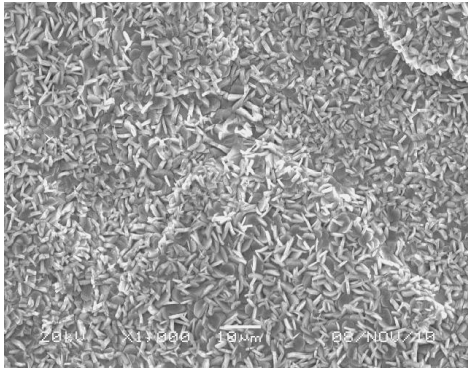
Aral Ropa 4093/HDG nr.3,  
normal and a spotted area, x1000



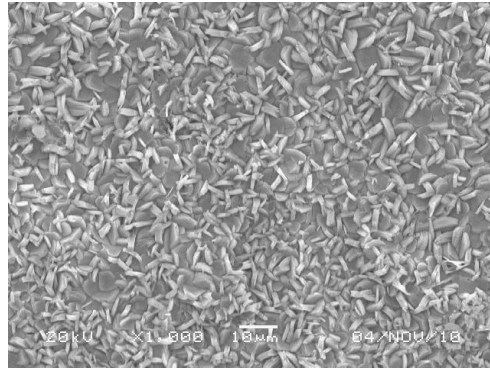
Multidraw KTL N16/HDG nr.1.1,  
normal area, x1000



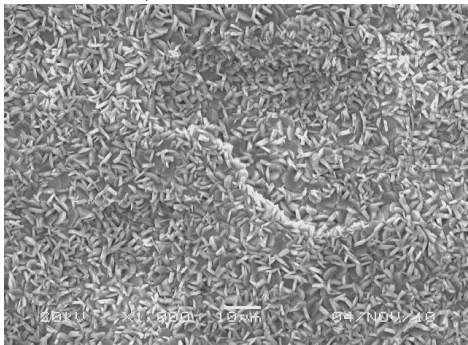
Multidraw KTL N16/HDG nr. 1.1,  
normal area, x1000



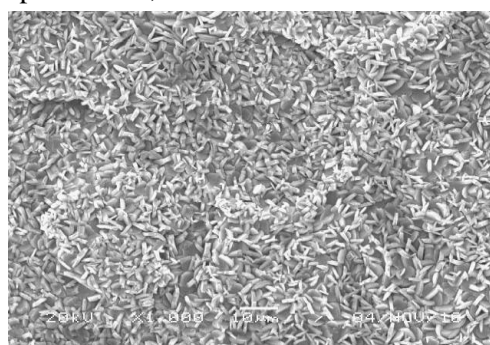
Multidraw KTL N16/HDG nr. 2.1,  
normal area, x1000



Multidraw KTL N16/HDG nr.2.1,  
spotted area, x1000

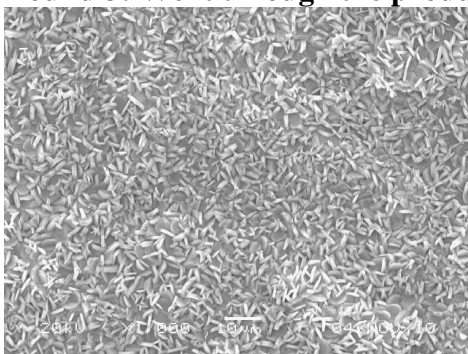


Iloform PL 6/HDG nr.2.1,  
normal area, x1000

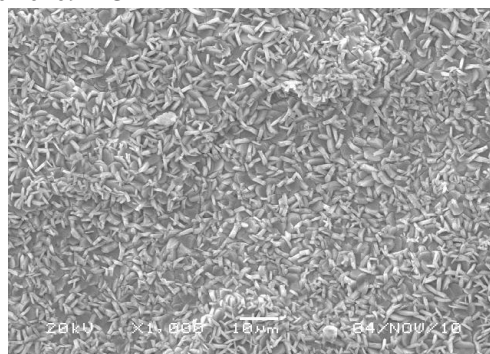


Iloform PL6/HDG nr. 1.1,  
normal area, x1000

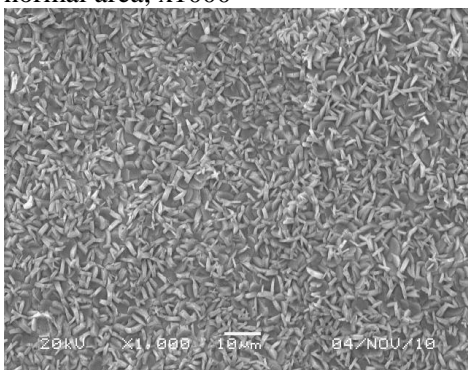
### Round 3: Went through the production 2010-09-23



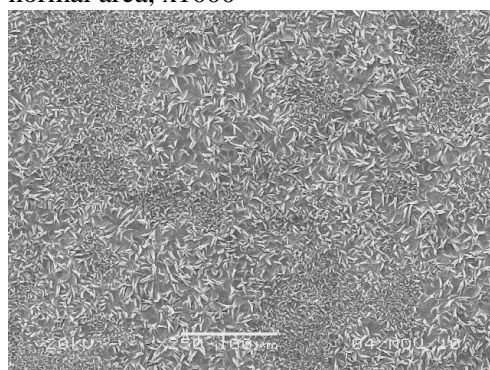
Anticorit RP 4107S/HDG nr 2,  
normal area, x1000



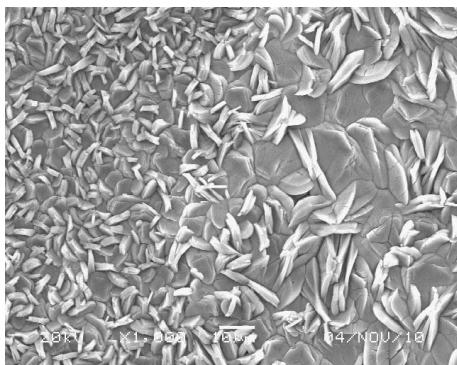
Anticorit RP 4107S/HDG nr 1,  
normal area, x1000



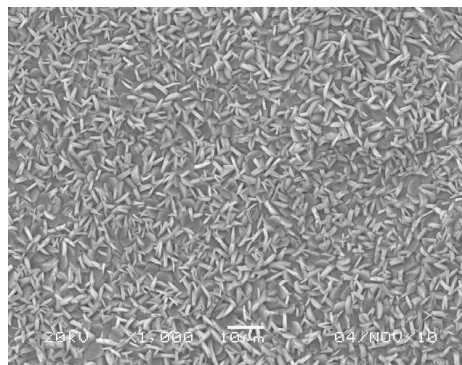
SW4020/HDG nr 1,  
normal area, x1000



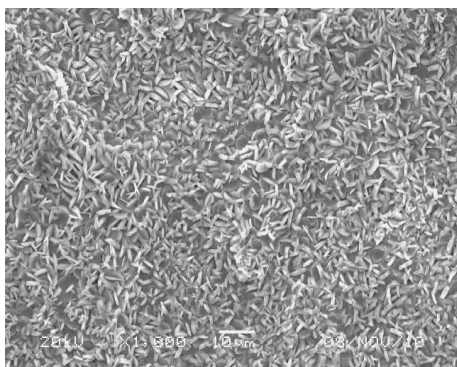
SW4020/HDG nr 2,  
spotted area, x250



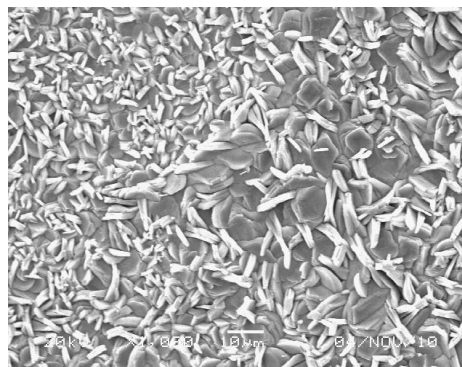
SW4020/HDG nr 2,  
normal and spotted area, x1000



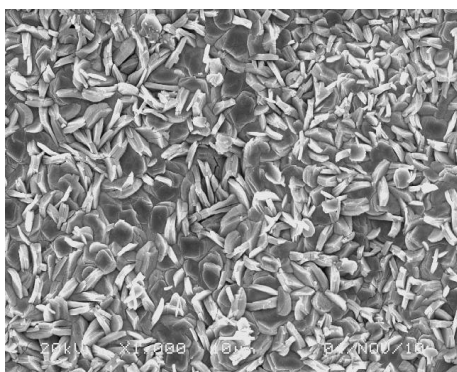
SW4020/HDG, nr 2,  
normal area, x1000



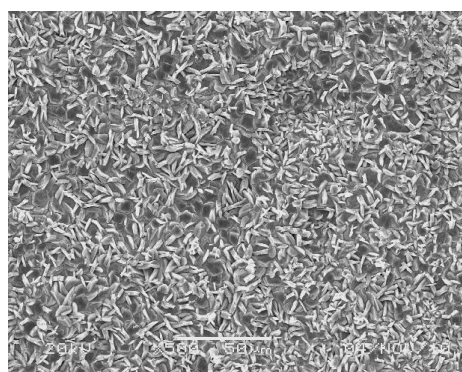
Binol Cut 10/HDG nr.2,  
normal area, x1000



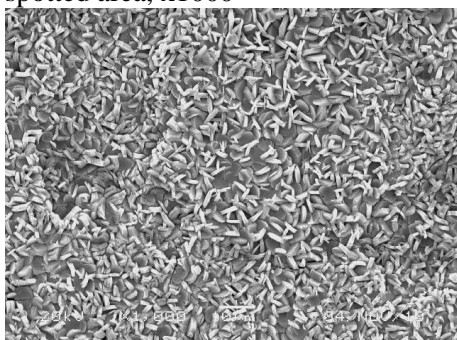
Binol Cut 10/HDG nr.2,  
spotted area, x1000



Binol Cut 10/HDG nr.3,  
spotted area, x1000

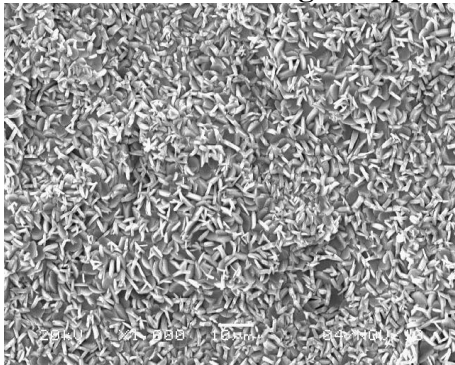


Binol Cut 10/HDG nr. 3,  
spotted area, x500

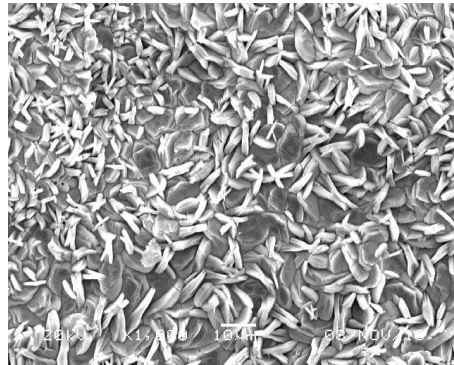


Binol Cut 10/HDG nr.3,  
normal area, x1000

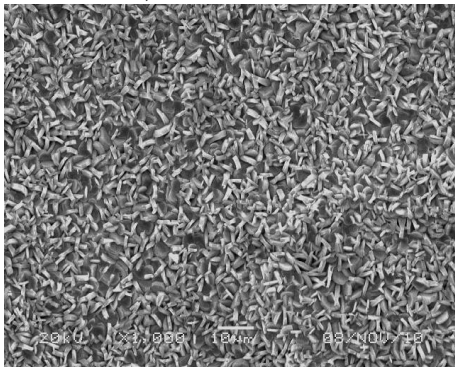
**Round 4: Went through the production 2010-09-28**



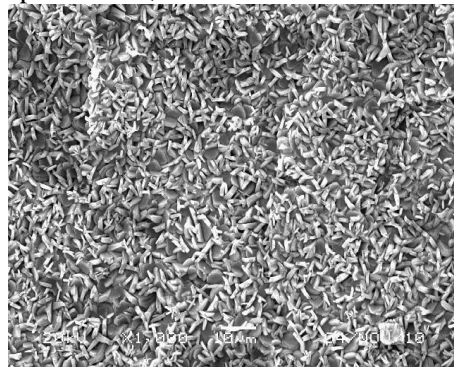
Multidraw KTL N16/HDG nr.1.2,  
normal area, x1000



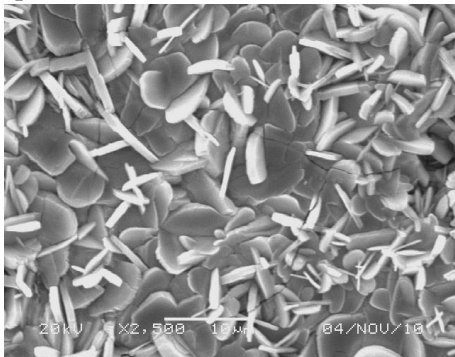
Multidraw KTL N16/HDG nr.2.2,  
spotted area, x1000



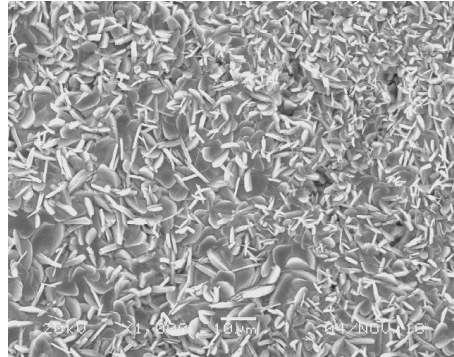
Multidraw KTL N16/HDG nr.2.2,  
spotted area, x1000



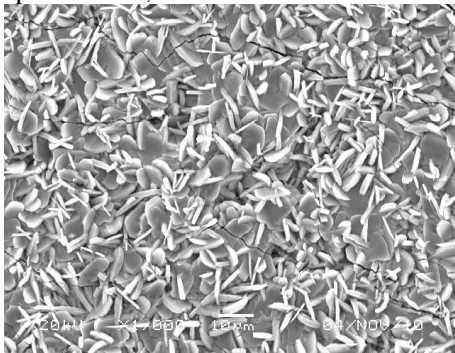
Multidraw KTL N16/HDG nr.2.2,  
normal area, x1000



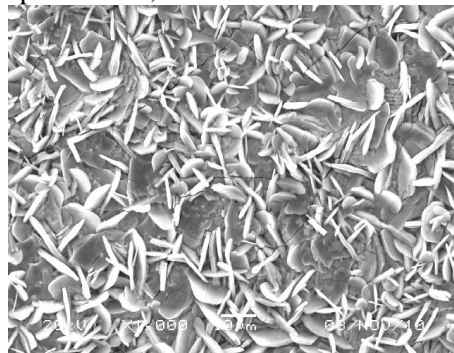
Binol Cut 20/GA nr.1,  
spotted area, x2500



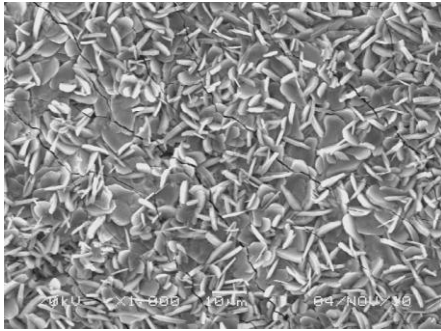
Binol Cut 20/GA nr.1,  
spotted area, x1000



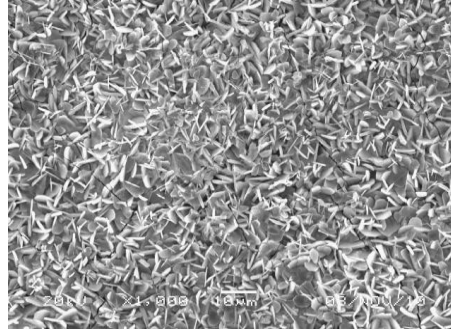
Binol Cut 20/GA nr.1,  
spotted area, x1000



Binol cut 20/GA nr 3,  
spotted area, x1000

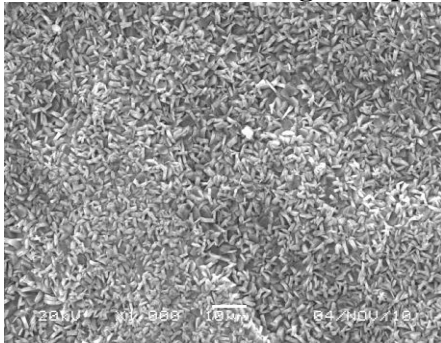


Binol Cut 20/GA nr.3,  
normal area, x1000

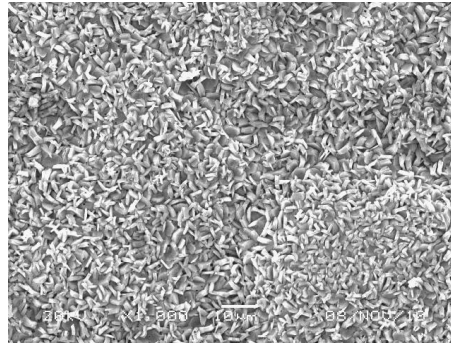


Binol Cut 20/GA nr.1,  
normal area, x1000

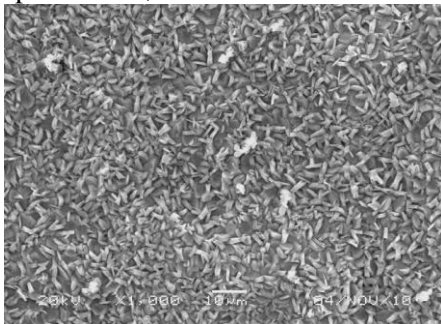
**Round 5: Went through the production 2010-09-30**



SW4020/EG nr.2,  
spotted area, x1000



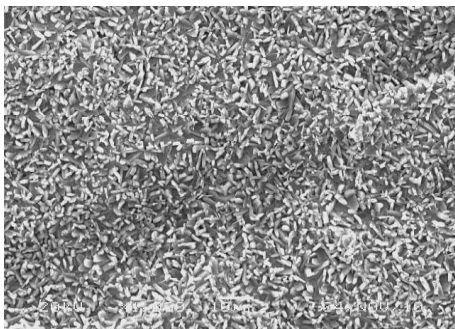
SW4020/EG nr.2,  
normal area, x1000



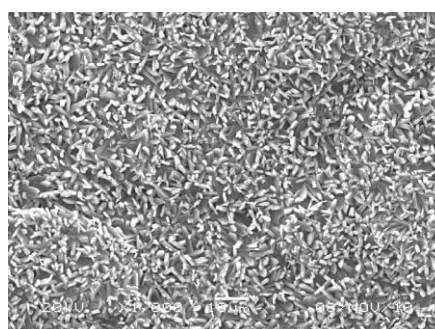
SW4020/EG nr.3,  
normal area, x1000

**Round 6: Went through the production 2010-10-06**

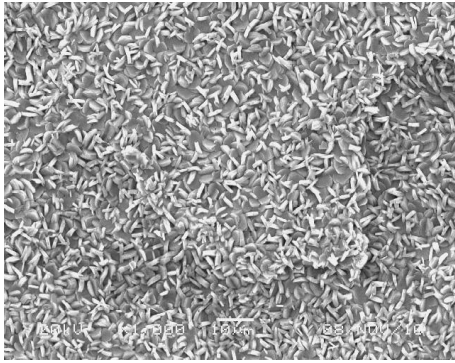
White dots - remark from production, nothing to do with this test.



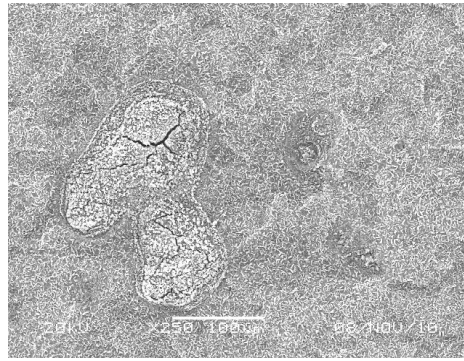
SW4020/CRS nr.1,  
normal area, x1000



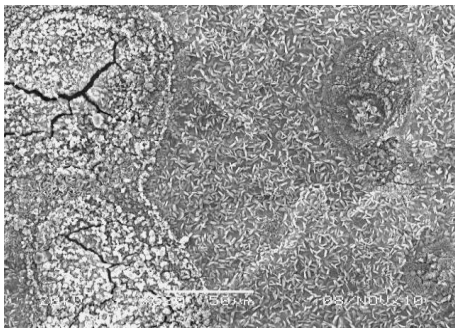
SW4020/CRS nr.2,  
normal area, x1000



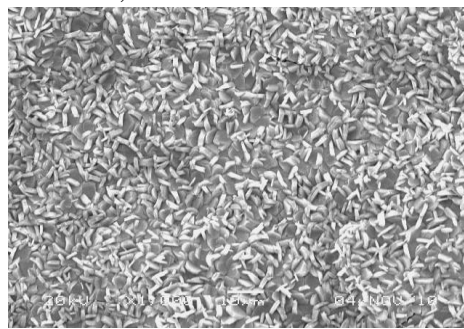
Umformöl 60/HDG nr.3,  
normal area, x1000



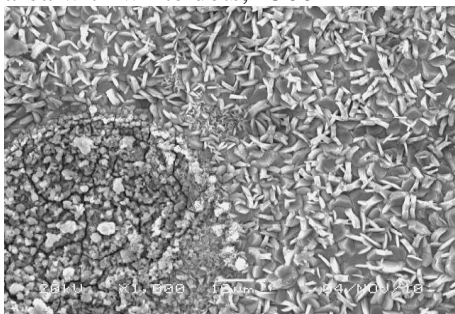
Umformöl 60/HDG nr.3  
normal area an area containing a  
white dot, x250



Umformöl 60/HDG nr.3  
area with white dots, x500



Umformöl 60/HDG nr.1,  
normal area, x1000



Umformöl 60/HDG nr1,  
area with white dots, x1000