

REPORT OF MIGRATION AT 23°C OF COMPOUNDS BASED IN 14944-3/2008 REGULATION FROM PRODUCTS USED TO BE IN CONTACT WITH WATER FOR HUMAN CONSUMPTION

Report Code: 3237801

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INDEX

1.	INTRODUCTION AND GENERAL INFORMATION	3
2.	ELEMENTS OF STUDY DESCRIPTION	4
3.	EXPERIMENTAL PROCEDURE	5
4.	RESULTS.	6
5.	CONCLUSIONS.	14
6.	ANNEXES	15



1. INTRODUCTION AND GENERAL INFORMATION.

The company KRYSTALINE TECHNOLOGY, S.A. has required a migration study of a solid material used to be in contact with water intended for human consumption.

LABORATORY DATA:

LABAQUA S.A.

Addres:

C/ Dracma, 16-18 Polígono Industrial Las Atalayas 03114 – Alicante España

Phone: 965 10 60 70 Fax: 965 10 60 80 info@labaqua.com

CLIENT DATA:

KRYSTALINE TECHNOLOGY, S.A. Address:
C/ ALFRED NOBEL, 18
03203-Elche/Elx
España



2. ELEMENTS OF STUDY DESCRIPTION.

Material pieces and application. The sample is a material intended to contain commercial water. Coating in cement plates coated by the customer.

Commercial name and batch: KRYSTALINE ADD1 Reception data in LABAQUA: 17/07/2020 10:54h Manufactured: KRYSTALINE TECHNOLOGY, S.A.

Samples remitted by: KRYSTALINE TECHNOLOGY, S.A.

Samples prepared by: KRYSTALINE TECHNOLOGY, S.A.

Assay pieces preparation: The samples consist of 10 reference concrete / mortar specimens (for the test blank) and 10 concrete / mortar specimens with Krystaline Add1 additive (for the sample) with the following dimensions:

Sample dimensions:

 $S_{Total} = 1.28 \text{ dm}^2$ each piece, 10 total pieces

Volume: 2L

Relación $S/V = 6.4 \text{ dm}^{-1}$



Figure 1. Picture of the test piece.



3. EXPERIMENTAL PROCEDURE.

This study is based on "UNE-EN ISO 14944-3: 2008 "Influencia de los materiales con base de cemento sobre el agua destinada al consumo humano, Método de ensayo. Parte 3: Migración de sustancias desde materiales con base de cemento" Regulation

When the migration water was obtained, the water sample was analyzed, indicated in the Annex I, in order to calculate the *migration rate*.

According with this Regulation and with CEN/CENELEC regulations, all the European countries are required to reach the specifications indicated.

Table 1. General information about the migration test:

Parameter	Data		
Total number of samples	20 pieces (10 pieces for the assay blank and 10 pieces for the sample with the additive)		
Final water volume	2 L		
Disinfection process	Not appropriate for this test		
Total surface area	1.28 dm ² /piece,		
S/V Ratio	6.4 dm ⁻¹		
Origin Water assay and temperature	Clorated water (1ppm) at 23.2±1°C for 72±1h,		
Test deviations	No deviations.		
Incidences	No incidences		
Static contact periods: 3 periods of 24h 1 period of 72h 1 period of 24h	03/08/2020 10:00h - 04/08/2020 10:00h 04/08/2020 10:00h - 05/08/2020 10:00h 05/08/2020 10:00h - 06/08/2020 10:00h 06/08/2020 10:00h - 09/08/2020 10:15h 09/08/2020 10:00h - 10/08/2020 10:00h		
Date and time of the start of the migration test	10/08/2020 10:00h — 13/08/2020 10:00h		
Date and time of the finish of the migration test	13/08/2020 10:00h		



Reagents used:

- Tap water from the laboratory.
- Chlorinated water with 1ppm of active chlorine
- Ordinary laboratory equipment
- Chronometer ID Plan 1873, ID Calibration 25126. (Laboratory code G-154)
- Bath of water ID Calibración 42667. Laboratory code (B-131)

Migration steps:

- <u>Sampling, shipment and preservation.</u> The sample was provided by the company KRYSTALINE TECHNOLOGY, S.A. No special preservation condition was required.
- <u>Sample preparation.</u> The client covered the cement sheets. The samples consist of 10 reference concrete / mortar specimens (for the test blank) and 10 concrete / mortar specimens with Krystaline Add1 additive (for the sample)
- <u>Pieces treatment.</u> The previous treatments consist on a washing step with a continuous water flow (rate between 1 to 3 m/min) during 60±5 minutes.
- <u>Static contact period</u>. The sheets were complete immersed in 2 liters of water during 3 periods of 24h, 1 period of 72h and 1 period of 24h. No disinfection process has been required.
- <u>Migration assay.</u> The sheets were immersed in 2L of Chlorinated water at 1ppm at 23±1°C for 72±1h. At the same time, a blank without samples pieces, have been also carried out in the same conditions than the samples.
- <u>Analysis</u>. The final concentration of the organic compounds was determined by means of the analytical methods indicated in the Annex I.

4. RESULTS.

Sample codification.

The Table 2 show the number of each analysis reports.



Table 2. Sample denomination.

	Code	Sample name	Total migration period (hours)
Analysis date 03/08/2020	5385453	CONTROL 10 P	72±1h
Analysis date 03/08/2020	5385454	KRYSTALINE ADD1	72±1h

Analytical results:

The final report with analytical results is in Annex I. Likewise the results of the Organic Compounds Analysis are in Annex II.

In the next Table the analytical results are showed.



5385453 (CONTROL 10 P)

Acrylamide	< 0.05	μg/L
Aldrin	< 0.01	μg/L
Aluminium	79	μg/L
Ammonium	< 0.10	mg/L
Antimony	< 2	μg/L
Arsenic	< 2	μg/L
Barium	10	μg/L
Screening of Organic Compounds BS EN 15768	Informe adjunto	
Benzo-(g,h,i)-perylene	< 0.01	μg/L
Benzo-a-pyrene	< 0.005	μg/L
Benzo-b-fluoranthene	< 0.01	μg/L
Benzo-k-fluoranthene	< 0.01	μg/L
Beryllium	< 2	μg/L
Bicarbonates	< 4.0	mg/L
Boron	< 10	μg/L
Bromates	< 10	μg/L
Cadmium	< 1	μg/L
Calcium	12.4	mg/L
Carbonates	24.9	mg/L
Total Organic Carbon (TOC)	< 0.5	mg/L
Total Cyanides	< 5	μg/L
Combined residual chlorine	< 0.05	mg/L
Free residual chlorine	1.04	mg/L
Total residual chlorine	1.03	mg/L
Vinyl chlorine	< 0.1	μg/L
Chloride	4.4	mg/L
Cobalt	< 2	μg/L
Cooper	< 2	μg/L
Colour	< 1.0	mg/L Pt/Co
Conductivity at 20°C	375	μS/cm
Chrome	< 2	μg/L



Dieldrin

Silver

Lead

Selenium

Sodium

Sulphates

Temperature
Turbidity

Vanadium

Zinc

Report 3237801

Epichlorohydrin < 0.10 μg/L Fluorides < 0.10 mg/L Heptachlor < 0.01 $\mu g/L$ Heptachlor epoxide < 0.01 $\mu g/L$ Iron < 10 $\mu g/L$ Indene-(1,2,3-c,d,)-pyrene < 0.01 μg/L Langelier's index 2.54 < 2 Manganese $\mu g/L$ < 0.20 Mercury $\mu g/L$ $\mu g/L$ Nickel < 2 < 0.5 **Nitrates** mg/L **Nitrites** < 0.05 mg/L Odor 1 Ind. de dil. Oxidability 0.37 $mg/O_2/L$ 11.2 U. pH. pН

03/09/2020

μg/L

 μ g/L

μg/L

μg/L

mg/L

mg/L

°C

UNF

μg/L

 $\mu g/L$

< 0.01

< 2

< 2

< 2

4.8

1.3

21.0

< 0.20

< 2

< 2



5385454 (KRYSTALINE ADD1)

Acrylamide	< 0.05	μg/L
Aldrin	< 0.01	μg/L
Aluminium	121	μg/L
Ammonium	< 0.10	mg/L
Antimony	< 2	μg/L
Arsenic	< 2	μg/L
Barium	15	μg/L
Screening of Organic Compounds BS EN 15768	Informe adjunto	
Benzo-(g,h,i)-perylene	< 0.01	μg/L
Benzo-a-pyrene	< 0.005	μg/L
Benzo-b-fluoranthene	< 0.01	μg/L
Benzo-k-fluoranthene	< 0.01	μg/L
Beryllium	< 2	μg/L
Bicarbonates	< 4.0	μg/L
Boron	< 10	μg/L
Bromates	< 10	μg/L
Cadmium	< 1	μg/L
Calcium	17.6	mg/L
Carbonates	23.0	mg/L
Total Organic Carbon (TOC)	< 0.5	mg/L
Total Cyanides	< 5	μg/L
Combined residual chlorine	< 0.05	mg/L
Free residual chlorine	0.95	mg/L
Total residual chlorine	0.99	mg/L
Vinyl chlorine	< 0.1	μg/L
Chloride	4.0	mg/L
Cobalt	< 2	μg/L
Cooper	< 2	μg/L
Colour	< 1.0	mg/L Pt/Co
Conductivity at 20°C	280	μS/cm
Chrome	< 2	μg/L



< 0.01 < 0.10 < 0.10 < 0.01 < 0.01 < 10 < 0.01 2.57	μg/L μg/L mg/L μg/L μg/L μg/L μg/L μg/L
< 0.10 < 0.01 < 0.01 < 10 < 0.01	mg/L μg/L μg/L μg/L
< 0.01 < 0.01 < 10 < 0.01	μg/L μg/L μg/L
< 0.01 < 10 < 0.01	μg/L μg/L
< 10 < 0.01	μg/L
< 0.01	
	μg/L
2.57	
2.07	
< 2	μg/L
< 0.20	μg/L
< 2	μg/L
< 0.5	mg/L
< 0.05	mg/L
1	Ind. de dil.
< 0.20	mg/ O ₂ /L
11.1	U. pH.
< 2	μg/L
< 2	μg/L
< 2	μg/L
3.3	mg/L
1.3	mg/L
21.2	°C
< 0.20	UNF
< 2	μg/L
< 2	μg/L
	< 2 < 0.20 < 2 < 0.5 < 0.05



Following the Regulation, the migration rate (M) was calculated with the next equation:

$$M = c_n / (S / V \cdot t) \text{ [mg dm}^{-2} \text{d}^{-1}\text{]}$$

where:

M = Migration rate

 c_n = The concentration obtained (expressed as mg/L and calculated with (c_n = a_n - b_n) where a_n is the concentration obtained in the migration and b_n the concentration obtained in the blank).

t = Migration period (3 days)

 $S/V = \text{rate surface / volume in dm}^{-1}$ (6.4 dm⁻¹)

Migration rate calculation:

Table 2.1. Results 5385454 at 23±2 °C.

COMPOUNDS ANALYZED				on and rate of ation
INCLUDED IN THE REPORT (ANNEX I)	b_n^T	a_n^T	${c_n}^T$	M_n^T
Acrylamide	< 0,05 µg/L	< 0,05 μg/L	< 0,05 μg/L	$< 2.60 \times 10^{-6}$ mg/dm ² día
Aldrin	< 0,01 µg/L	< 0,01 µg/L	< 0,01 μg/L	$< 5.21 \times 10^{-6}$ mg/dm ² día
Aluminium	79 μg/L	121 μg/L	42 μg/L	2.19 ×10 ⁻³ mg/dm ² día
Ammonium	< 0.10 mg/L	< 0.10 mg/L	< 0.10 mg/L	$< 5.21 \times 10^{-3}$ mg/dm ² día
Antimony	< 2 μg/L	< 2 μg/L	< 2 μg/L	$< 1.04 \times 10^{-4}$ mg/dm ² día
Arsenic	< 2 μg/L	< 2 μg/L	< 2 μg/L	$ < 1.04 \times 10^{-4} $ $ mg/dm^2 día $
Barium	10 μg/L	15 μg/L	5 μg/L	2.60 ×10 ⁻⁴ mg/dm ² día
Benzo–(g,h,i)– perylene	< 0,01 µg/L	< 0,01 µg/L	< 0,01 μg/L	$< 5.21 \times 10^{-6}$ mg/dm ² día



:port 3237801	T		1	03/03/2020
Benzo-a-pyrene	< 0,005 μg/L	< 0,005 μg/L	< 0,005 μg/L	$< 2.60 \times 10^{-7}$ $mg/dm^2 dia$
Benzo-b- fluoranthene	< 0,01 μg/L	< 0,01 µg/L	< 0,01 μg/L	$< 5.21 \times 10^{-6}$ mg/dm ² día
Benzo-k- fluoranthene	< 0,01 μg/L	< 0,01 µg/L	< 0,01 μg/L	$< 5.21 \times 10^{-6}$ mg/dm ² día
Beryllium	< 2 μg/L	< 2 μg/L	< 2 μg/L	$< 1.04 \times 10^{-4}$ mg/dm ² día
Bicarbonates	< 4,0 mg/L	< 4,0 mg/L	< 4,0 mg/L	$< 2.08 \times 10^{-1}$ mg/dm ² día
Boron	< 10 μg/L	< 10 μg/L	< 10 μg/L	$< 5.21 \times 10^{-4}$ mg/dm ² día
Bromates	< 10 μg/L	< 10 μg/L	< 10 μg/L	$< 5.21 \times 10^{-4}$ mg/dm ² día
Cadmium	< 1 μg/L	< 1 μg/L	< 1 μg/L	$< 5.21 \times 10^{-5}$ mg/dm ² día
Calcium	12,4 mg/L	17,6 mg/L	5,2 mg/L	2.71 ×10 ⁻¹ mg/dm ² día
Carbonates	24,9 mg/L	23,0 mg/L	< 2,0 mg/L	1.04×10^{-1} mg/dm ² día
Total Organic Carbon (TOC)	< 0,5 mg/L	< 0,5 mg/L	< 0,5 mg/L	$< 2.60 \times 10^{-2}$ mg/dm ² día
Total Cyanides	< 5 μg/L	< 5 μg/L	< 5 μg/L	$< 2.60 \times 10^{-4}$ mg/dm ² día
Vinyl chlorine	< 0,1 μg/L	< 0,1 μg/L	< 0,1 μg/L	$< 5.21 \times 10^{-5}$ $mg/dm^2 dia$
Chlorine	4,4 mg/L	4,0 mg/L	< 1,0 mg/L	$< 5.21 \times 10^{-2}$ $mg/dm^2 dia$
Cobalt	< 2 μg/L	< 2 μg/L	< 2 μg/L	$< 1.04 \times 10^{-4}$ mg/dm ² día
Cooper	< 2 μg/L	< 2 μg/L	< 2 μg/L	$< 1.04 \times 10^{-4}$ mg/dm ² día
Chrome	2 μg/L	< 2 μg/L	< 2 μg/L	$< 1.04 \times 10^{-4}$ mg/dm ² día
Dieldrin	< 0,01 μg/L	< 0,01 µg/L	< 0,01 μg/L	$< 5.21 \times 10^{-6}$ mg/dm ² día



Sulphates

Vanadium

Zinc

Screening of

Organic Compunds

Report 3237801

< 5.21 ×10⁻⁵ Epichlorohydrin $< 0.10 \mu g/L$ $< 0.10 \ \mu g/L$ $< 0.10 \, \mu g/L$ mg/dm² día < 5.21 ×10⁻⁶ Heptachlor $< 0.01 \, \mu g/L$ $< 0.01 \mu g/L$ $< 0.01 \mu g/L$ mg/dm² día < 5.21 ×10⁻⁶ Heptachlor epoxide $< 0.01 \mu g/L$ $< 0.01 \mu g/L$ $< 0.01 \, \mu g/L$ mg/dm² día < 5.21 ×10⁻⁴ Iron $< 10 \mu g/L$ $< 10 \mu g/L$ $< 10 \mu g/L$ mg/dm² día < 5.21 ×10⁻⁶ Indene-(1,2,3-c,d,)- $< 0.01 \mu g/L$ $< 0.01 \mu g/L$ $< 0.01 \, \mu g/L$ mg/dm² día pyrene < 1.04 × 10⁻⁴ Manganese $< 2 \mu g/L$ $< 2 \mu g/L$ $< 2 \mu g/L$ mg/dm² día $< 1.04 \times 10^{-5}$ Mercury $< 0.20 \mu g/L$ $< 0.20 \mu g/L$ $< 0.20 \mu g/L$ mg/dm² día < 1.04 × 10⁻⁴ Nickel $< 2 \mu g/L$ $< 2 \mu g/L$ $< 2 \mu g/L$ mg/dm² día < 1.04 × 10⁻⁴ Silver $< 2 \mu g/L$ $< 2 \mu g/L$ $< 2 \mu g/L$ mg/dm² día < 1.04 ×10⁻⁴ Lead $< 2 \mu g/L$ $< 2 \mu g/L$ $< 2 \mu g/L$ mg/dm² día < 1.04 × 10⁻⁴ Selenium $< 2 \mu g/L$ $< 2 \mu g/L$ $< 2 \mu g/L$ mg/dm² día < 5.21×10⁻² Sodium 4,8 mg/L < 1.0 mg/L3,3 mg/Lmg/dm² día < 5.21×10⁻²

1,3 mg/L

 $< 2 \mu g/L$

 $2 \mu g/L$

 $< 10,0 \mu g/L$

< 1.0 mg/L

 $< 2 \mu g/L$

 $< 2 \mu g/L$

 $< 10.0 \, \mu g/L$

 $\frac{\text{mg/dm}^2 \text{ día}}{< 1.04 \times 10^{-4}}$

 $\frac{\text{mg/dm}^2 \text{ día}}{< 1.04 \times 10^{-4}}$

 $\frac{\text{mg/dm}^2 \text{ día}}{< 5.21 \times 10^{-4}}$

mg/dm² día

03/09/2020

1,3 mg/L

 $< 2 \mu g/L$

 $< 2 \mu g/L$

 $< 10,0 \mu g/L$



5. CONCLUSIONS.

- The final liquid obtained in the migration process at 23±2°C in the test water has been analyzed in order to determine some compounds included and indicated in table 2.1 and calculated the migration rate.
- In the sample codified as 5385454 KRYSTALINE ADD1 migration of metals like Aluminum, Barium and Calcium, were detected. In the case of Aluminum, does not exceed the value set by the RD-140/2003 which establishes a maximum value of $200\mu g/L$. In the case of Barium and Calcium, there is not a maximum value set by the RD-140/2003.
- The product codified as 5385454 (KRYSTALINE ADD1) meets all the criteria set out in RD 140/2003 Regulation, for use in materials with drinking water.



6. ANNEX

ANNEX I. Analysis report for the samples 5385453 and 5385454.

ANNEX II. Screening of organic compounds.