TEST METHOD

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NEUTRAL SALT SPRAY TEST

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REFERENCED DOCUMENT

External Standard : NF EN 10152 (1994).

FOREWORD

The results of the work carried out in France within the framework of a work group gathering salt spray users of various origins (CBBS) are integrated in the test method

- *Motor-vehicle manufacturers,*
- *Chamber builders,*
- *Surface treatment product formulators,*
- *cold immersion or electrolytic zinc coating applicators,*
- *CETIM.*

PREAMBLE

There is rarely a direct relationship between the resistance to salt spray and the resistance to corrosion in other environments since the corrosion products are different. Therefore, the test results should not be regarded as a direct indication of the resistance to corrosion of metal materials in all the environments in which they may be used. Under no circumstances is this test a method for the study of the resistance to salt sprays. Also, the behaviour of the various materials during the test should not be regarded as an indication of their relative resistance to corrosion in commission.

The test which is described gives a method to check the quality of the materials and coatings with respect to determined specifications.

1. FIELD OF APPLICATION

This method is used to evaluate the resistance to corrosion of metal materials protected by a permanent or temporary coating.

In general, the corrosion test under neutral salt spray conditions applies to metal materials protected by (anodic and cathodic) metal coatings, conversion coatings, or organic coatings.

This method has been drawn up specifically for coatings which are less noble than steel, and which are deposited on a ferrous substrate, such as allied zinc and zinc electrodeposits, or zinc alloy deposits obtained with cold immersion, with or without conversion and/or reinforced finish.

However, a chamber used and checked under the method conditions shall comply in every respect with the recommendations of the standards mentioned in the text. It will be possible to use it to test all coating types. For instance: for coatings which are nobler than steel, and which are deposited on a ferrous substrate, organic coatings, or conversion coatings, the salt spray test makes it possible to detect porosities, discontinuities, or adherence defects (case of paints).

No rules are prescribed in this method as to the dimensions of the parts to be tested, the exposure periods for a particular product, or the interpretation of the results. These details are given in the corresponding product specifications.

2. PRINCIPLE

The salt spray test consists in a corrosion attack accelerated by an artificial salt spray with a defined composition under accurate temperature and pressure conditions.

The degree of corrosion is evaluated:

- either with the appearance of corrosion spots,
- or with the percentage of corroded surface,
- or with damage noted by means of a micrographic examination,
- or with changes in the mechanical properties.

3. APPARATUS

The material or the coating of all the parts coming into contact with the spray or the test solution shall withstand the corrosion due to the sprayed solution, and shall have no influence on its aggressiveness. The testing apparatus includes the following items:

3.1. SPRAY CHAMBER

The minimum capacity of the spray chamber shall be 0,4 m^3. For high-volume chambers, the spray shall be distributed uniformly irrespective of the chamber condition (empty or loaded). The upper part of the chamber shall be designed in such a way that accumulated solution drops, which run on the surface, cannot flow off the tested parts. The walls and the cover shall be isolated to ensure homogeneity of the temperature and of the size of the spray droplets in the entire volume of the chamber.

The dimensions and the shape of the spray chamber shall be such that the quantity of solution collected in each collecting basin is within the limits of 1 ml/h to 2 ml/h, for an approx. 80 cm^2 horizontal recovery area (78,5 cm^2, i.e. 10-cm diameter). The collecting basins used for the test shall never be installed under the parts being tested.

The condensed solution is evacuated at the bottom of the chamber.

3.2. TEMPERATURE REGULATING AND HEATING DEVICE

A suitable device should make it possible to maintain the test chamber and what it contains at a temperature of 35 °C ± 2 °C, and to avoid spray condensation phenomena on the walls of the chamber to the maximum. The measurements shall be taken at a minimum distance of 100 mm from the walls. The temperature shall be checked at least on each working day; continuous monitoring is recommended.

3.3. SPRAYING DEVICE

The salt solution feeding device consists of a system for clean air, the pressure and humidity of which are checked, feeding, a tank containing the solution to be sprayed, and of one or several sprayers.

The compressed air shall be filtered so that it contains no impurities (oil, dust); the allowed maximum is 0,2 mg/m^3 for a size < 5 µm. The pressure shall be between 0,8 bar and 1,6 bar according to the manufacturer's recommendations. Spray distribution in the test chamber is promoted by a high pressure.

The compressed air shall be saturated with water to prevent an increase in the mass concentration of the chloride in the sprayed solution during pressure reduction. The air is humidified before coming into the sprayer by flowing through an air humidifier containing water heated to a temperature greater than the temperature prevailing in the chamber (45 °C – 47°C). The equipment shall ensure air saturation. The water column shall be filled, and its height shall be kept at a constant level, with demineralised water (conductivity < 20 µS/cm). The air humidifier shall be fitted with a thermometer to measure the water temperature, and a discharge hole making it possible to sample water in order to check its conductivity.

The suitable temperature for the air from the air humidifier depends on the pressure used and on the spraying nozzle.

The quality of the air shall be checked indirectly by monitoring the air humidifier water conductivity. If the latter changes beyond 20 µS/cm, the air humidifier shall be drained, cleaned, and it will be necessary to improve the quality of the air.

Recommendation: the temperature and the conductivity of the air humidifier water can be recorded usefully in a daily monitoring document on chamber operation.

The sprayers shall be manufactured from materials that are inert with respect to the salt solution. Their configuration shall be adapted to the chamber configuration; the assembly shall be designed so that the spray is distributed uniformly at all points in the useful volume of the chamber. The term "useful volume" designates the area in which the quantity of collected solution and aggressiveness comply, i.e. that they are within the limits prescribed in paragraph 4.1. If the chamber is designed correctly, the total volume is useful.

On chamber models with an internal reserve of salt solution, the level of the solution in this tank (placed inside the spray chamber) shall be kept constant to ensure a regular spray feeding throughout the test.

3.4. COLLECTING BASINS

The collecting basins are used at least during yearly checks and monthly monitorings.

Use funnels of diameter 10 cm, consisting of smooth glass, (opening surface equal to approx. 80 cm^2) placed on glass gauges as collecting devices. The constituent materials of the collecting basin assembly shall be made from chemically inert materials.

Depending on the test (see annex 1), at least 1 collecting basin for a 300-litre chamber placed near the reference plates is necessary.

4. SALT SOLUTION

4.1. PREPARATION

The conductivity of the water shall be equal to or less than 20 µS/cm at 20 °C ± 2 °C

In distilled water or de-mineralised water, dissolve the necessary quantity of sodium chloride to obtain a concentration of 50 g/l ± 5 g/l.

In the anhydrous condition, the sodium chloride shall neither contain a total of more than 0,2 % (m/m) of impurities, nor more than 0,1 % (m/m) of sodium iodide. It shall be free of copper and nickel (less than 10 mg/kg for each of the two components).

The density of the salt solution, measured at 20 °C ± 2 °C, shall be between 1,025 and 1,040.

4.2. pH MEASUREMENT

The pH shall be measured at 20 °C ± 2 °C with a pH-meter with a highly alkaline electrode. The electrode shall not touch the bottom of the beaker. The solution shall be moderately stirred. The Ph shall be read after 1 min.

The pH of the salt solution which is prepared shall not be corrected. It shall be between 6,5 and 7,2 after a maximum 6-hour stabilisation time. If this is not the case, the solution cannot be used.

4.3. FILTERING

The solution shall be transparent (observation with the naked eye); if necessary, filter it before it is poured in the device tank.

5. METHODS IMPLEMENTED TO CHECK AND MONITOR TEST CHAMBER AGGRESSIVENESS

It is necessary to check the aggressiveness of the test chamber to control the reliability and the reproducibility of the results obtained in a chamber, or make a comparison between the results from various chambers.

THE METHOD WHICH IS IMPLEMENTED TO CHECK AND MONITOR THE AGGRESSIVENESS OF THE TEST CHAMBER is appended in annex 1: it defines the parameters of the reference plates, in particular:

- characteristics (chemical composition, coating thickness...),
- cleaning mode before the test,
- protection mode for the parts which are not subjected to the test,
- number and position of the plates inside the chamber,
- method implemented to evaluate the corroded surface,
- determination of the "mark" or degree of aggressiveness.

The aggressiveness shall be CHECKED in an empty chamber at least once every year, and prior to returning to service after maintenance work or important repairs carried out on the equipment.

The aggressiveness shall be MONITORED every month under the filling conditions that the current use requires.

6. TEST PIECE AND PART PREPARATION

6.1. The number, type, shape, and dimensions of the test pieces and of the parts to be tested shall be chosen in accordance with the rules for the specification of the material or product being tested. Failing such a specification, they shall be the subject of an agreement between the parties concerned.

6.2. The test pieces and the polluted parts (grease...) shall be cleaned carefully before the test. The method used depends on the type of material, on the surface, and on the products which have contaminated this surface; use of abrasives or solvents which may attack the surface of the part shall not be included in it.

Make sure that the test pieces and the parts are not contaminated again after cleaning with careless handling.

The test pieces and parts covered intentionally with greasy protective films (oils, greases…) shall not be cleaned before the test..

6.3. If the test pieces and the parts to be tested are cut from larger coated parts, this shall be done so as not to damage the coating in the area which is adjacent to the cut. Unless otherwise specified, the cutting edges shall be protected in a suitable manner with the appropriate coating, which cannot be attacked under the test conditions, such as wax, paint, or adhesive tape.

6.4. The salt spray test shall only be performed after a minimum period of 24 hours depending on the treatment made.

6.5. If the test pieces and the parts to be tested should undergo preliminary oven drying before the corrosion test (case of certain automotive specifications), the following conditions shall apply:

- oven drying shall take place at least 24 hours after deposit,
- it will be possible to perform the salt spray test as soon as the test pieces and the parts have returned to the ambient temperature after oven drying.

7. LAYOUT OF TEST PIECES AND PARTS TO BE TESTED

7.1. The test pieces and the parts shall be placed in the chamber in such a way that they are not in the direct way of the spray.

7.2. The angle under which the surface is exposed in the chamber is very important. The test piece or the part to be tested shall be placed in the spray chamber with the side to be characterised upward forming a 20° ± 5° angle with the vertical. In the case of parts with irregular shapes, this recommendation shall be taken into account as far as possible, and the rating shall be limited to the surface which is properly oriented.

7.3. The test pieces and the parts to be tested shall be arranged in such a way as not to come into contact with the chamber. The test surfaces to be characterised shall be exposed to the free flow of the spray. The parts may be placed at various levels in the spray chamber, provided that the spray does not run from the parts or mountings located at higher exposure levels.

Exchanging test pieces and parts is recommended whenever a daily check is performed.

7.4. The constituent materials of the test piece stands and of the parts used to hang them shall be inert with respect to the salt solution and the parts to be tested. The test pieces and the parts shall be held fixed throughout the test. In the special case of fasteners, it is recommended to immobilise the parts with fitting (screwing) onto suitable supporting plates.

8. OPERATING CONDITIONS

8.1. The test can only be started after the conformity of the chamber aggressiveness has been CHECKED / MONITORED beforehand (annexe 1).

8.2. The temperature inside the test chamber shall be maintained at 35 °C ± 2 °C at all points measured at more than 10 cm from the wall.

8.3. The pressure and spraying shall be kept constant in the chamber during the test time.

8.4. The average rate for recovery of the solution by the collecting basin shall be measured on each working day. It shall be equal to 1 ml/h to 2 ml/h for an approx. 80 cm^2 horizontal area. It is advisable to use at least one collecting basin placed halfway between the sprayer and the chamber wall which is the farthest.

8.5. The sprayed test solution shall not be reused.

8.6. The records for the test conditions shall be entered in the DAILY MONITORING REPORT (annex 2.1).

9. TEST DURATION

9.1. The test duration shall be set in accordance with the rules for the specification of the material or product being tested. Failing such a specification, it shall be the subject of an agreement between the parties involved.

9.2. Process control: the end of the test depends on the corrosion appearance. The parts shall be checked daily without rinsing.

9.3. Product control: the end of the test depends on the expiration of a determined period; a visual examination of the parts subjected to the test may be performed daily. The orientation of the parts shall not be modified, and the total chamber opening time shall not exceed one hour per day. The total chamber opening time is not deducted from the part exposure time in the chamber.

10. TEST PIECE AND PART TREATMENT ON TEST COMPLETION

At the end of the test, take the test pieces and the parts out of the chamber, and remove the residue of sprayed solution deposited on the surface with care. For this purpose, the parts may be rinsed or immersed in running water at a temperature not exceeding 37 °C, then dried immediately in a current of warm air.

11. EXPRESSION OF RESULTS

The test results can be appraised according to many different criteria depending on the special requirements given in the specifications of the material or product being tested:

- presence of white rust (corrosion products of: zinc, zinc alloys, zinc-based coatings, aluminium, magnesium, …),
- presence of red rust (ferrous substrate corrosion products),
- time for the initial corrosion products to appear,
- visual rating of the corroded surface or of the corrosion pit density / unit area,
- variation in the weight or volume,
- damage noted with a micrographic examination,
- modification of the mechanical properties.

12. TRAINING

The chamber operators shall have been given a training which includes the following, in particular: information on the various standards; operating principle of a chamber; preparation of a salt solution; salt solution pH measurement; chamber maintenance and check; chamber aggressiveness check and monitoring; method implemented to appraise the result; and drafting of test reports. When training is complete, the trainee will identify the various types of corrosion which may appear in a salt spray chamber.

13. TEST REPORT

Information on the following shall be recorded in it:

- description of the material or of the parts to be tested,
- reference of the reference standard (material specification),
- reminder of the requirements for the product being tested,
- number of test pieces and parts subjected to the test,
- chamber aggressiveness mark,
- date of the last yearly and/or monthly aggressiveness check, and date of the check to come,
- test duration and results of intermediate checks, if any,
- anomalies and incidents which occurred during the tests,
- average result obtained for all the parts of the batch being tested,
- if necessary, photograph of the parts being tested.

NOTE: Two typical reports are presented in annexes 1.5 and 2.2.

These two documents are applicable within the framework of work conducted on zinc-based coatings.

ANNEX 1

AGGRESSIVENESS CHECK AND MONITORING METHOD

INTRODUCTION

The objective of this protocol is to check the aggressiveness of the test chamber in a salt spray chamber intended to evaluate the performance level of a zinc-based coating. It sets parameters identified as critical in surveys conducted within the framework of CBBS, determines the conditions under which the chamber or its settings are regarded as compliant, and defines an aggressiveness level. The latter will be expressed as a mark and indicated in the test report.

Two procedures are applicable:

The purpose of the AGGRESSIVENESS CHECK is to check the operation of the chamber over the entire useful volume independently of any test performed on a part.

The purpose of the AGGRESSIVENESS MONITORING is to keep track of the chamber performance between two CHECKING periods.

The test chamber aggressiveness checking and monitoring protocol refers to:

- results of tests performed within the framework of CBBS,
- the test method.

NOTE: The reference plates used in this method are not connected with the international metrology.

1. PERIODICITY

The AGGRESSIVENESS shall be CHECKED at least once a year, and prior to returning to operation, after maintenance work or important repairs on the equipment.

The AGGRESSIVENESS shall be MONITORED at least once a month.

2. OPERATING CONDITIONS

All the parameters set in the test method shall be complied with.

2.1. REFERENCE PLATES

The reference plates shall consist of DC 04 type steel according to Standard NF EN 10152, coated (at least on one side) with a zinc layer obtained by continuous high speed on line zinc electroplating.

The zinc thickness shall be 10 µm ± 1 µm. It shall be possible to cover the plates with an oil film intended to ensure better preservation.

The plates shall be accompanied by a certificate of conformity which shall include:

- the Supplier's identification,
- the product identification: coil No and casting No,
- the chemical and mechanical characteristics of the supporting metal,
- the thickness of the zinc deposit,
- the reference of the protection oil.

ANNEX 1 (CONTINUED)

2.2. SALT SOLUTION PREPARATION

The salt solution shall be prepared in accordance with the test method.

2.3. PREPARATION OF REFERENCE PLATES

The reference plates shall be degreased in the following way:

1. Preliminary degreasing with acetone using a soft cloth.
2. Ultrasonic degreasing in a washing bath with the following composition:

.	Sodium hydrogen carbonate	($NaHCO_3$)	15 g/l	± 2 g/l
.	Sodium carbonate	(Na_2CO_3)	10 g/l	± 2 g/l
.	Trisodium phosphate	(Na_3PO_4)	20 g/l	± 2 g/l
.	Borax	($Na_2B_4O_7$, $10H_2O$)	10 g/l	± 2 g/l
.	De-mineralised water		q.s.p 1 litre.	

Conditions of use with application of ultrasonics:

.	Temperature	45° C (± 2° C)
.	Time	7 min. (± 1min).

This washing bath is usable for 5 plates per litre.

3. With ultrasonics application, take the plate out with tweezers, rinse it under mains water, then with methanol, and allow it to dry in the air.

2.4. MASK

The degreased plates shall be handled with gloves. The edges of the plates and the back shall be protected with an adhesive tape, 100 mm x 38 mm, tobacco brown type, according to the drawing given in annex 1.1.

2.5. POSITION OF REFERENCE PLATES AND COLLECTING BASINS

The stand shall be manufactured from a chemically inert material. It shall allow positioning of the plates according to a checked angle of 20 ° (± 5 °) with respect to the vertical. The centre of the plate shall be at sprayer height. The number and position of the plates varies depending on the test and the chamber design (annexe 1.2):

- at least 3 during a YEARLY CHECK. They shall be placed at a quarter, one half, and three quarters of the distance between the sprayer and the chamber wall which is the farthest, with the side to be corroded facing the sprayer,
- at least 1 during a MONTHLY MONITORING. The plate shall be placed halfway between the sprayer and the chamber wall which is the farthest.

The collecting basins shall be placed according to paragraph 4.4. in the test method.

ANNEX 1 (CONTINUED)

2.6. CHAMBER FILLING

The chamber shall only contain the reference plates during the ANNUAL CHECK.

During the MONTHLY MONITORING, the test is performed under the filling conditions that the current use requires. The other parts which are exposed shall not shelter the reference plate.

2.7. CORRODED SURFACE EVALUATION

On a tracing, reproduce the measurement mask given in annex 1.1. Place the mask on the reference plate. Consider that a square is oxidised as soon as there is red rust on it (including runs).

Checking is done visually every 24 hours on a plate which is not rinsed, still humid.

Checking is done from 72 hours. Starting the test on Friday is recommended.

If there is red rust on one of the plates during the check on reaching 72 hours, it is necessary to resume the test on Monday and to carry out a check every 24 hours.

In the case of the annual check, observe a 30 min/day opening time from 72 hours.

In the case of the monthly monitoring, each opening shall not exceed 60 min. The opening time is not deducted.

2.8. EXPRESSION OF RESULTS

TRR (Temps de Rouille Rouge: Red Rust Time) designates the time that has elapsed before a rusting level greater than à 5 % is observed for the first time (i.e. 6 squares at least with red rust).

The degree of aggressiveness of the chamber is evaluated with a mark from 1 to 5.

TRR (hours)	Mark	Conformity
< or = 72	1	Does not comply
from 72 to 96	2	**Complies**
from 96 to 120	3	
from 120 to 144	4	
> 144	5	Does not comply

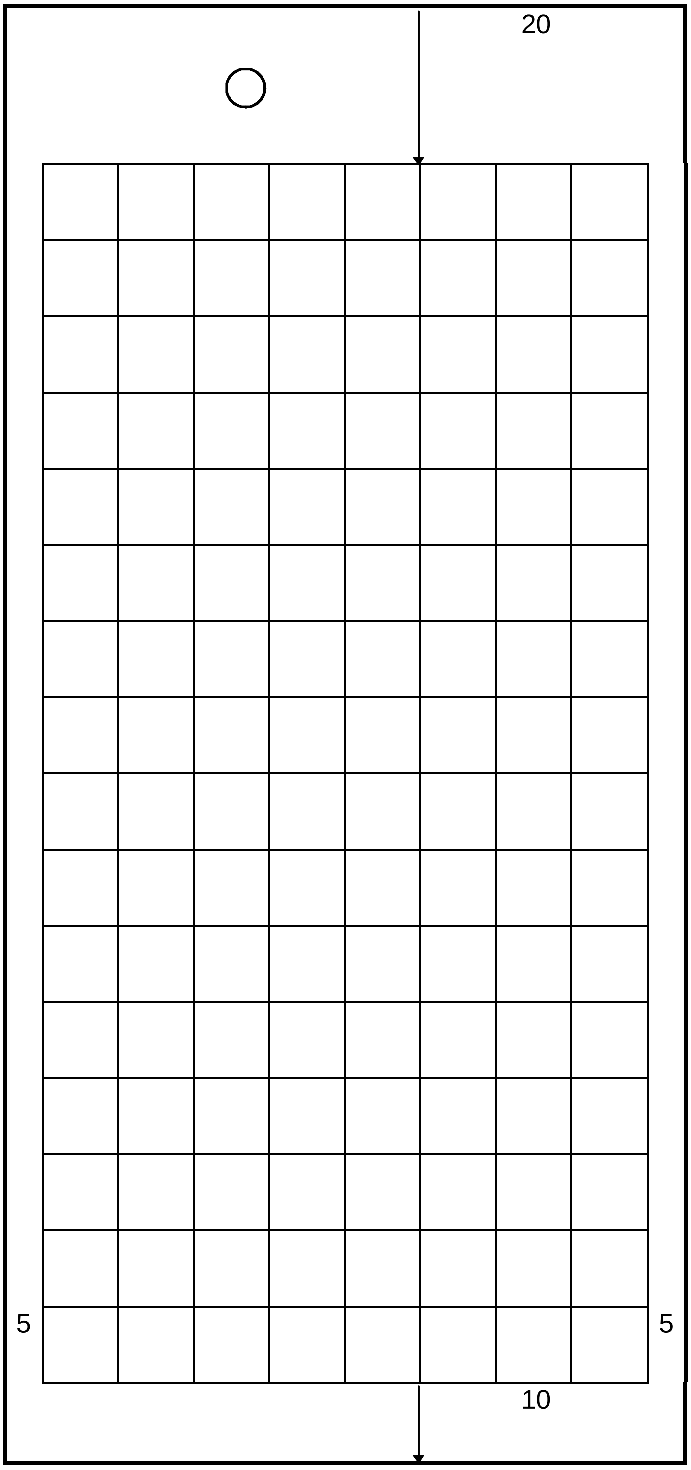
A chamber complies if the mark = 2, 3 or 4.

3. INSPECTION REPORT

The typical report given in annex 1.3 shall be fully completed.

ANNEX 1.1

MASK DRAWING / MASK FOR CONTROL OF REFERENCE PLATES



Zinc electroplated plate 190 mm X 90 mm

Surface of a square = 1 cm^2

% corroded = n squares X 0,78

Total exposed surface = 128 cm^2

ANNEX 1.2

REFERENCE PLATE POSITION PLAN

Chamber with centred sprayer

Type of test:	Annual check ☐	Monthly monitoring ☐
Test starting date …………..		Chamber identification number ………

CONTROL OF CONDENSATION WATER

	Test piece 1	Test piece 2	Test piece 3
Collected volume, in ml / h∗			

∗ Average measurement over the entire test duration (including opening time)

Note: the number of the test piece corresponds to the number of the plate being tested.

AGGRESSIVENESS EVALUATION

		Appearance of red rust: % recorded on control mask		
TRR	Mark	Plate 1	Plate 2	Plate 3
= 48 hours	1			
72 hours	2			
96 hours	3			
120 hours	4			
=144 hours	5			
Result (Mark)				

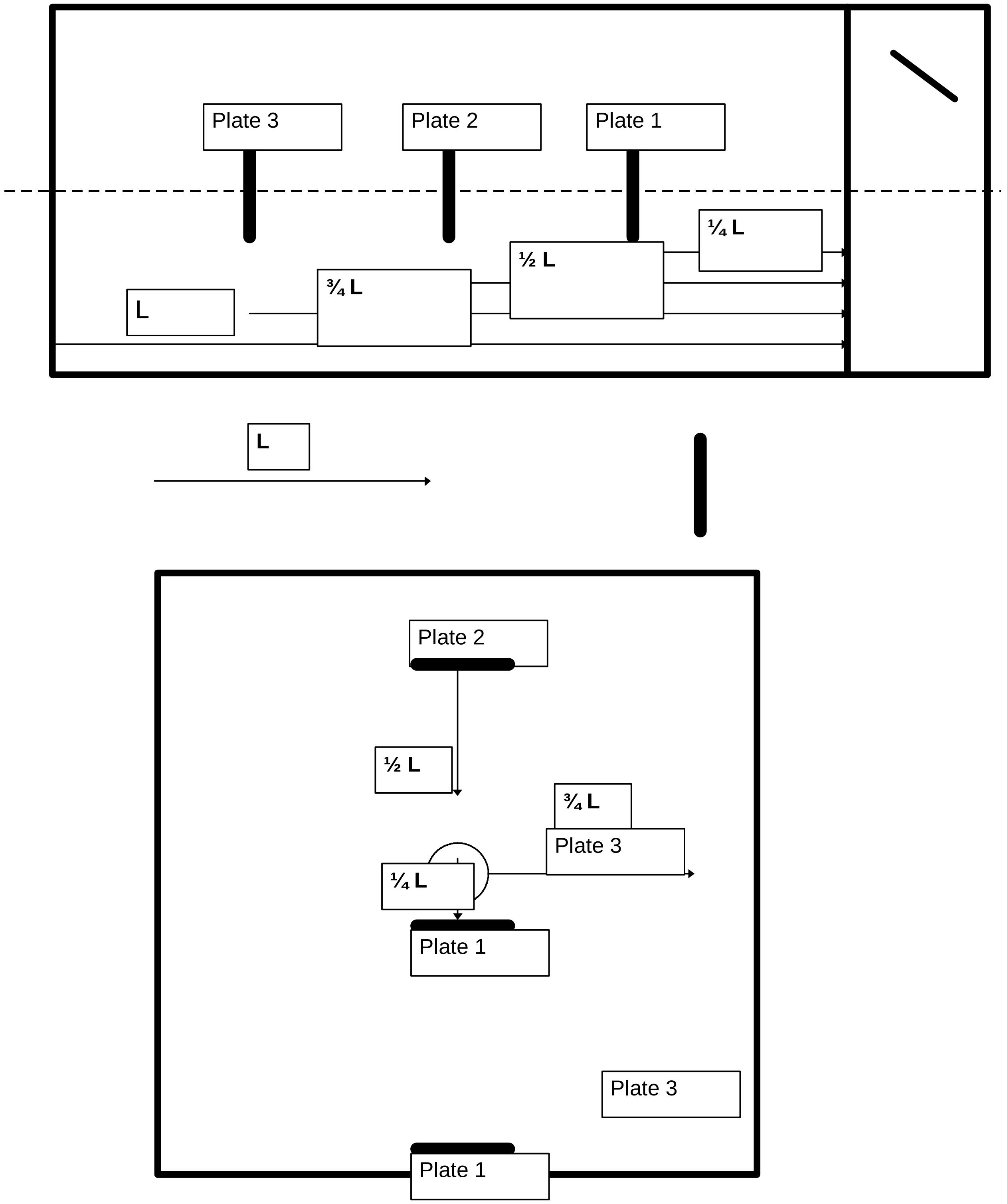
CONCLUSION OF AGGRESSIVENESS AND CONDENSATION WATER CONTROL

COMPLIES ALL PARAMETERS ☐	**DOES NOT COMPLY** ☐	
Comments		
Operator	Signature	Date

ANNEX 1.3

TEST CHAMBER AGGRESSIVENESS CHECKING AND MONITORING REPORT

Chamber with off-centre sprayer



ANNEX 2.1

DAILY MONITORING REPORT

Year:			Month:			Operator:	
Date	Running total	Air	Initial salt solution		Test chamber		Comments
		Pressure 0.8 to 1.6 bars	pH 6.5 to 7.2	Density 1025 to 1040 Kg/m^3	Temperature 33 to 37°C	Volume of condensation water 1 to 2 ml/hour	
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
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ANNEX 2.2

TEST REPORT

IDENTIFICATION

Test No:……………. Salt spray chamber No …………………..

Customer's name: . . . . Order / BATCH No: . . . .

Reference / Designation : . . . . BATCH quantity: . . . .

Type of coating: . . . . Applicator: . . . .

Line designation : . . . .

Demanded thickness: . . . . µm Special condition: ……………………………..

Thickness found: . . . . .µm Oven drying: h °C

TEST CHAMBER AGGRESSIVENESS MONTHLY CHECK

Date of last check: . . . . Date of next check: . . . .

AGGRESSIVENESS MARK (1 to 5): . . . . (complies between 2 and 4)

TEST PARAMETERS

Starting date: . . . . Number of parts: . . . .

Requirements: White rust . . . . Hours Red rust . . . . Hours

TEST RESULTS

During each observation, indicate the number of parts involved. The sum of each column should correspond to the total number of parts for the test

A: no rust **B**: appearance of white rust **C**: white rust over more than 5% of the surface **D**: appearance of red rust

C																					
B																					
A																					
Hours	24	48	72	96	120	144	168	192	216	240	264	288	312	336	360	384	408	432	456	480	504
C																					
B																					
A																					
hours	528	552	576	600	624	648	672	696	720	744	768	792	816	840	864	888	912	936	960	984	1008

Appearance of White Rust	**Appearance of Red Rust**	**Total test duration**
. . . . Hours	**. . . . . Hours**	**. . . . . Hours**

TEST CONCLUSION

COMPLIES	**DOES NOT COMPLY. .. . . (%)**

Comments:

Operator	Signature	Date