# TECHNICAL VALIDATION OF NEW ANALYTICAL TOOLS INTENDED FOR THE QUALITY CONTROL OF CORK STOPPERS

## DETERMINATION OF THE MOISTURE CONTENT OF NATURAL CORK STOPPERS BY MICROWAVE HYGROMETER *WAVECORK*

**FINAL REPORT**

**CTCOR TEST DOSSIER No.: 5669/19**

**Client:**
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Santa Maria de Lamas, November 2019

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## 1. INTRODUCTION AND OBJECTIVES

The aim of present study is to evaluate and validate a new analytical system intended for the determination of cork stoppers' moisture content, developed by EGITRON - Engenharia e Automação Industrial, Lda., under the trademark Microwave Hygrometer *WaveCork*.

For this purpose, it was investigated the correlation between the cork stoppers moisture content obtained by direct reading on Microwave Hygrometer *WaveCork* and the moisture content determined on these same cork stoppers by using the reference method established in NP ISO 9727-3:2011 - *Cylindrical cork stoppers. Physical tests. Part 3: Determination of humidity content*.

At the same time, and at the request of the Client, the above results were compared with the moisture values obtained by a direct reading on a needle conductivity meter, a device currently used by cork industry's operators for the quality control of cork stoppers.

## 2. DESCRIPTION OF THE EQUIPMENT UNDER VALIDATION

Equipment trade name: Wave Cork

Brand: EGITRON

Model: WCK-2019-01

Serial Number: 000 (prototype)

Indication Range: (0 – 10) %

Resolution: 0,1 %

Summary description (*Text provided by the Client*):

WaveCork has been developed to determine the moisture content present in cork stoppers, by means of an accurate, non-contact and non-destructive procedure.

Its principle of operation consists in the emission and reception of microwaves within a resonant cavity in which the cork stopper is placed. The correlation between the gain and the respective frequency range allows to estimate the moisture content in a whole-body quantification, not limited to a single zone and with a response speed of 3-4 seconds.

The system was developed towards Industry 4.0 paradigm, presenting communication capacity via MQTT protocol (IIOT) for integration with other subsystems of the production chain.

Due to the lack of uniformity in water distribution within cork stoppers, the expedited moisture measurement method commonly used in the cork industry presents large variations in the measured value. *WaveCork* proposes to eliminate these reading variation factors, bringing more reliability and robustness to the results presented.

## 3. DESCRIPTION OF ACESSORY EQUIPMENT, USED FOR COMPARISON

Equipment trade name: *Aqua-boy hygrometer*

Brand: KPM

Model: Aqua Boy KOM II

Indication Range: (5 – 20) %

Resolution: 0,25 %

## 4. TASKS PLAN

In order to achieve the outlined objectives, the following tasks were defined and implemented:

Task 1 - Sampling of cylindrical natural cork stoppers, of nominal size 45x24 mm and with a quality visual grade approximated to 1º/2º (according to CTCOR's visual grade standards). These cork stoppers were submitted to essay under washed and uncoated condition.

Task 2 - Conditioning of the cork stoppers within climatic chambers / rooms, in order to obtain moisture values in the entire range of the equipment indication.

Task 3 - Determination of the moisture content of the cork stoppers by direct reading on the measuring equipments (*Wavecork* and *Aquaboy*).

Task 4 - Determination of the moisture content of the cork stoppers by applying the reference method NP ISO 9727-3:2011.

Task 5 - Data processing, critical analysis of the results and elaboration of final report.

## 5. TEST METHODOLOGY

In the following of the present study, the cork stoppers were conditioned in order to obtain moisture contents ranging from 0% to 10%, thus allowing to carry out the verification over the entire indication range of the *WaveCork* equipment.

After conditioning, and for each specimen under test, immediate readings were carried out on the microwave hygrometer *WaveCork* and on the *Aquaboy* hygrometer. Afterwards, the same cork stoppers were weighed in an analytical balance. Subsequent analytical tasks were performed in accordance with the *long method* test methodology defined in NP ISO 9727-3:2011.

## 6. RESULTS

The following tables and graphs show the values obtained from the different measurements made on a same specimen (cork stopper), based on the following three methodologies:

i) direct reading on the *WaveCork* microwave hygrometer;
ii) reading on the *Aquaboy* hygrometer, followed by mathematical adjustment applying the characteristic correlation curve of the equipment in use;
iii) Reference method (gravimetric, after constant weight drying), as laid down in test standard NP ISO 9727-3:2011.

*Note: Correlation test carried out on a total of 80 natural cork stoppers, corresponding to 80 correlation points.*

**Table 1. Determination of moisture content in cork stoppers. Individual results obtained by applying the reference method, by a direct reading on the microwave hygrometer *WaveCork* and by a correlated reading on the *Aquaboy* hygrometer.**

***Legend**: **mi** - cork stopper's initial mass, expressed in (g); **mf** - cork stopper's final mass, expressed in (g);*
***Reference** - cork stopper's moisture content as obtained by applying the methodology defined in NP ISO 9727-3:2011;*
***Aquaboy** - cork stopper's moisture content as obtained by a direct reading on the Aquaboy hygrometer;*
***Aquaboy corr.** - cork stopper's moisture content as obtained by reading on the Aquaboy hygrometer, followed by mathematical adjustment applying the characteristic correlation curve of the equipment;*
***WaveCork** - cork stopper's moisture content as obtained by a direct reading on the WaveCork microwave hygrometer.*

| Point no. | Cork stopper's mass (g) | | Moisture content (%) | | | | Aquaboy corr. error | WaveCork error |
|---|---|---|---|---|---|---|---|---|
| | mi | mf | Reference | Aquaboy | Aquaboy corr. | WaveCork | | |
| 5 | 3,2032 | 3,1168 | 2,7 | | | 3,1 | | 0,4 |
| 3 | 3,7300 | 3,6257 | 2,8 | | | 3,3 | | 0,5 |
| 2 | 3,5990 | 3,4905 | 3,0 | | | 3,4 | | 0,4 |
| 1 | 3,5396 | 3,4437 | 2,7 | | | 3,4 | | 0,7 |
| 4 | 3,7146 | 3,5970 | 3,2 | | | 3,8 | | 0,6 |
| 14 | 3,0387 | 2,8920 | 4,8 | 6,1 | 5,9 | 4,7 | 1,1 | -0,1 |
| 8 | 2,9276 | 2,7790 | 5,1 | 5,9 | 5,7 | 4,8 | 0,6 | -0,3 |
| 12 | 3,3555 | 3,1755 | 5,4 | 6,0 | 5,8 | 5,0 | 0,4 | -0,3 |
| 11 | 3,6240 | 3,4544 | 4,7 | 5,3 | 5,1 | 5,2 | 0,4 | 0,5 |
| 6 | 3,2867 | 3,1228 | 5,0 | 5,6 | 5,4 | 5,2 | 0,4 | 0,2 |
| 10 | 3,2084 | 3,0447 | 5,1 | 5,5 | 5,3 | 5,2 | 0,2 | 0,1 |
| 13 | 3,6266 | 3,4596 | 4,6 | 5,3 | 5,2 | 5,3 | 0,6 | 0,6 |
| 7 | 3,9275 | 3,7193 | 5,3 | 5,7 | 5,5 | 5,5 | 0,2 | 0,1 |
| 18 | 3,1119 | 2,9444 | 5,4 | 6,3 | 6,0 | 5,6 | 0,6 | 0,2 |
| 15 | 3,5356 | 3,3511 | 5,2 | 5,8 | 5,5 | 5,6 | 0,3 | 0,4 |
| 19 | 2,7531 | 2,5845 | 6,1 | 6,8 | 6,6 | 5,7 | 0,5 | -0,4 |
| 26 | 3,1977 | 3,0125 | 5,8 | 6,0 | 5,8 | 5,8 | 0,0 | 0,0 |
| 24 | 3,0746 | 2,9079 | 5,4 | 6,0 | 5,8 | 5,8 | 0,4 | 0,4 |
| 9 | 4,0497 | 3,8234 | 5,6 | 5,7 | 5,5 | 5,9 | -0,1 | 0,3 |
| 23 | 3,4493 | 3,2457 | 5,9 | 7,5 | 7,5 | 6,0 | 1,6 | 0,1 |
| 94 | 3,0365 | 2,8589 | 5,8 | 5,9 | 5,7 | 6,1 | -0,2 | 0,2 |
| 27 | 3,2612 | 3,0631 | 6,1 | 6,6 | 6,4 | 6,1 | 0,3 | 0,0 |
| 96 | 3,1239 | 2,9597 | 5,3 | 6,1 | 5,9 | 6,1 | 0,6 | 0,8 |
| 93 | 2,6601 | 2,4932 | 6,3 | 6,5 | 6,2 | 6,1 | 0,0 | -0,2 |
| 28 | 3,7477 | 3,5399 | 5,5 | 6,0 | 5,8 | 6,1 | 0,2 | 0,6 |
| 97 | 3,2057 | 2,9987 | 6,5 | 5,9 | 5,7 | 6,5 | -0,8 | 0,0 |
| 95 | 3,2766 | 3,0601 | 6,6 | 6,1 | 5,9 | 6,5 | -0,7 | -0,1 |
| 17 | 3,2051 | 2,9762 | 7,1 | 6,7 | 6,5 | 6,5 | -0,6 | -0,6 |
| 20 | 3,6885 | 3,4544 | 6,3 | 6,0 | 5,8 | 6,6 | -0,6 | 0,3 |
| 36 | 3,2691 | 3,0433 | 6,9 | 7,6 | 7,6 | 6,7 | 0,7 | -0,2 |
| 29 | 3,4897 | 3,2382 | 7,2 | 7,8 | 7,8 | 6,7 | 0,6 | -0,5 |
| 21 | 3,5529 | 3,2757 | 7,8 | 6,9 | 6,7 | 6,8 | -1,1 | -1,0 |
| 22 | 3,8596 | 3,6039 | 6,6 | 7,2 | 7,0 | 7,0 | 0,4 | 0,4 |
| 31 | 3,4871 | 3,2214 | 7,6 | 7,1 | 7,0 | 7,2 | -0,6 | -0,4 |

*(continues)*

**Table 1** (cont.). **Determination of moisture content in cork stoppers. Individual results obtained by applying the reference method, by a direct reading on the microwave hygrometer *WaveCork* and by a correlated reading on the *Aquaboy* hygrometer.**

***Legend***: ***mi*** *- cork stopper's initial mass, expressed in (g);* ***mf*** *- cork stopper's final mass, expressed in (g);*
***Reference*** *- cork stopper's moisture content as obtained by applying the methodology defined in NP ISO 9727-3:2011;*
***Aquaboy*** *- cork stopper's moisture content as obtained by a direct reading on the Aquaboy hygrometer;*
***Aquaboy corr.*** *- cork stopper's moisture content as obtained by reading on the Aquaboy hygrometer, followed by mathematical adjustment applying the characteristic correlation curve of the equipment;*
***WaveCork*** *- cork stopper's moisture content as obtained by a direct reading on the WaveCork microwave hygrometer.*

| Point no. | Cork stopper's mass (g) | | Moisture content (%) | | | | Aquaboy corr. error | WaveCork error |
|---|---|---|---|---|---|---|---|---|
| | mi | mf | Reference | Aquaboy | Aquaboy corr. | WaveCork | | |
| 16 | 3,5089 | 3,2351 | 7,8 | 6,5 | 6,3 | 7,2 | -1,5 | -0,6 |
| 25 | 4,4389 | 4,1172 | 7,2 | 6,6 | 6,4 | 7,4 | -0,9 | 0,1 |
| 33 | 3,1917 | 2,9165 | 8,6 | 8,0 | 8,1 | 7,4 | -0,5 | -1,2 |
| 39 | 3,2679 | 3,0070 | 8,0 | 8,2 | 8,3 | 7,5 | 0,4 | -0,5 |
| 44 | 2,9723 | 2,7214 | 8,4 | 7,7 | 7,7 | 7,5 | -0,8 | -0,9 |
| 98 | 3,9352 | 3,6888 | 6,3 | 7,4 | 7,3 | 7,5 | 1,0 | 1,3 |
| 60 | 3,3924 | 3,1129 | 8,2 | 8,6 | 9,0 | 7,6 | 0,8 | -0,6 |
| 34 | 3,8582 | 3,5646 | 7,6 | 8,0 | 8,2 | 7,6 | 0,6 | 0,0 |
| 30 | 3,8307 | 3,5315 | 7,8 | 7,8 | 7,8 | 7,7 | 0,0 | -0,1 |
| 41 | 3,0105 | 2,7520 | 8,6 | 7,9 | 8,0 | 7,8 | -0,6 | -0,8 |
| 64 | 3,1040 | 2,8100 | 9,5 | 9,1 | 9,8 | 7,8 | 0,3 | -1,6 |
| 52 | 3,4686 | 3,1980 | 7,8 | 8,6 | 9,0 | 7,9 | 1,2 | 0,1 |
| 47 | 3,1912 | 2,9161 | 8,6 | 8,0 | 8,1 | 8,0 | -0,5 | -0,6 |
| 32 | 4,2038 | 3,8544 | 8,3 | 8,2 | 8,3 | 8,0 | 0,0 | -0,3 |
| 55 | 3,4882 | 3,2050 | 8,1 | 8,2 | 8,3 | 8,0 | 0,2 | -0,1 |
| 48 | 3,8806 | 3,5815 | 7,7 | 8,0 | 8,1 | 8,1 | 0,4 | 0,4 |
| 59 | 3,6230 | 3,3466 | 7,6 | 8,8 | 9,2 | 8,2 | 1,6 | 0,5 |
| 42 | 3,5602 | 3,2322 | 9,2 | 7,5 | 7,5 | 8,2 | -1,7 | -1,0 |
| 63 | 3,4986 | 3,2033 | 8,4 | 9,6 | 10,6 | 8,3 | 2,2 | -0,2 |
| 45 | 3,6646 | 3,3470 | 8,7 | 7,6 | 7,6 | 8,4 | -1,1 | -0,3 |
| 99 | 4,0061 | 3,6579 | 8,7 | 7,3 | 7,2 | 8,4 | -1,4 | -0,3 |
| 65 | 3,3572 | 3,0386 | 9,5 | 9,2 | 10,0 | 8,4 | 0,5 | -1,1 |
| 50 | 3,3164 | 3,0205 | 8,9 | 9,0 | 9,6 | 8,4 | 0,7 | -0,5 |
| 100 | 3,6677 | 3,3302 | 9,2 | 7,8 | 7,8 | 8,4 | -1,4 | -0,8 |
| 37 | 4,6507 | 4,2606 | 8,4 | 7,0 | 6,8 | 8,6 | -1,5 | 0,2 |
| 75 | 3,8364 | 3,5081 | 8,6 | 9,3 | 10,2 | 8,6 | 1,6 | 0,0 |
| 82 | 2,9478 | 2,6365 | 10,6 | 10,1 | 11,6 | 8,7 | 1,1 | -1,9 |
| 40 | 3,9429 | 3,5913 | 8,9 | 7,8 | 7,8 | 8,8 | -1,1 | -0,1 |
| 57 | 3,5166 | 3,1697 | 9,9 | 8,6 | 9,0 | 8,8 | -0,8 | -1,1 |
| 70 | 3,3569 | 3,0216 | 10,0 | 10,1 | 11,6 | 8,8 | 1,6 | -1,2 |
| 49 | 3,7793 | 3,4462 | 8,8 | 8,7 | 9,1 | 8,8 | 0,3 | 0,0 |
| 46 | 4,3805 | 3,9245 | 10,4 | 7,5 | 7,5 | 9,9 | -2,9 | -0,5 |

*(continues)*

**Table 1** (cont.). **Determination of moisture content in cork stoppers. Individual results obtained by applying the reference method, by a direct reading on the microwave hygrometer *WaveCork* and by a correlated reading on the *Aquaboy* hygrometer.**

***Legend**: **mi** - cork stopper's initial mass, expressed in (g); **mf** - cork stopper's final mass, expressed in (g);*
***Reference** - cork stopper's moisture content as obtained by applying the methodology defined in NP ISO 9727-3:2011;*
***Aquaboy** - cork stopper's moisture content as obtained by a direct reading on the Aquaboy hygrometer;*
***Aquaboy corr.** - cork stopper's moisture content as obtained by reading on the Aquaboy hygrometer, followed by mathematical adjustment applying the characteristic correlation curve of the equipment;*
***WaveCork** - cork stopper's moisture content as obtained by a direct reading on the WaveCork microwave hygrometer.*

| Point no. | Cork stopper's mass (g) | | Moisture content (%) | | | | Aquaboy corr. error | WaveCork error |
|---|---|---|---|---|---|---|---|---|
| | mi | mf | Reference | Aquaboy | Aquaboy corr. | WaveCork | | |
| 67 | 3,4835 | 3,1310 | 10,1 | 9,5 | 10,5 | 8,9 | 0,3 | -1,2 |
| 35 | 5,3001 | 4,8322 | 8,8 | 7,0 | 6,8 | 9,2 | -2,0 | 0,4 |
| 73 | 3,4244 | 3,0678 | 10,4 | 9,3 | 10,2 | 9,2 | -0,2 | -1,2 |
| 43 | 4,3179 | 3,8883 | 9,9 | 7,4 | 7,3 | 9,3 | -2,7 | -0,6 |
| 76 | 3,8409 | 3,4471 | 10,3 | 9,9 | 11,1 | 9,3 | 0,9 | -0,9 |
| 66 | 3,7713 | 3,3551 | 11,0 | 9,5 | 10,5 | 9,4 | -0,5 | -1,6 |
| 69 | 3,8302 | 3,4055 | 11,1 | 9,5 | 10,5 | 9,5 | -0,6 | -1,6 |
| 51 | 4,0512 | 3,6529 | 9,8 | 9,0 | 9,7 | 9,5 | -0,2 | -0,3 |
| 53 | 4,0112 | 3,6003 | 10,2 | 8,8 | 9,3 | 9,6 | -0,9 | -0,7 |
| 62 | 4,3662 | 3,9440 | 9,7 | 8,8 | 9,3 | 9,6 | -0,4 | 0,0 |
| 89 | 3,8738 | 3,4220 | 11,7 | 10,5 | 12,5 | 9,8 | 0,8 | -1,9 |
| 54 | 3,7607 | 3,3702 | 10,4 | 8,6 | 9,0 | 9,8 | -1,4 | -0,6 |
| 71 | 4,6409 | 4,1967 | 9,6 | 9,5 | 10,5 | 9,8 | 1,0 | 0,3 |
| 85 | 3,5187 | 3,1049 | 11,8 | 9,8 | 11,0 | 9,9 | -0,8 | -1,9 |
| 83 | 3,7945 | 3,3526 | 11,6 | 10,3 | 12,0 | 10,0 | 0,4 | -1,6 |

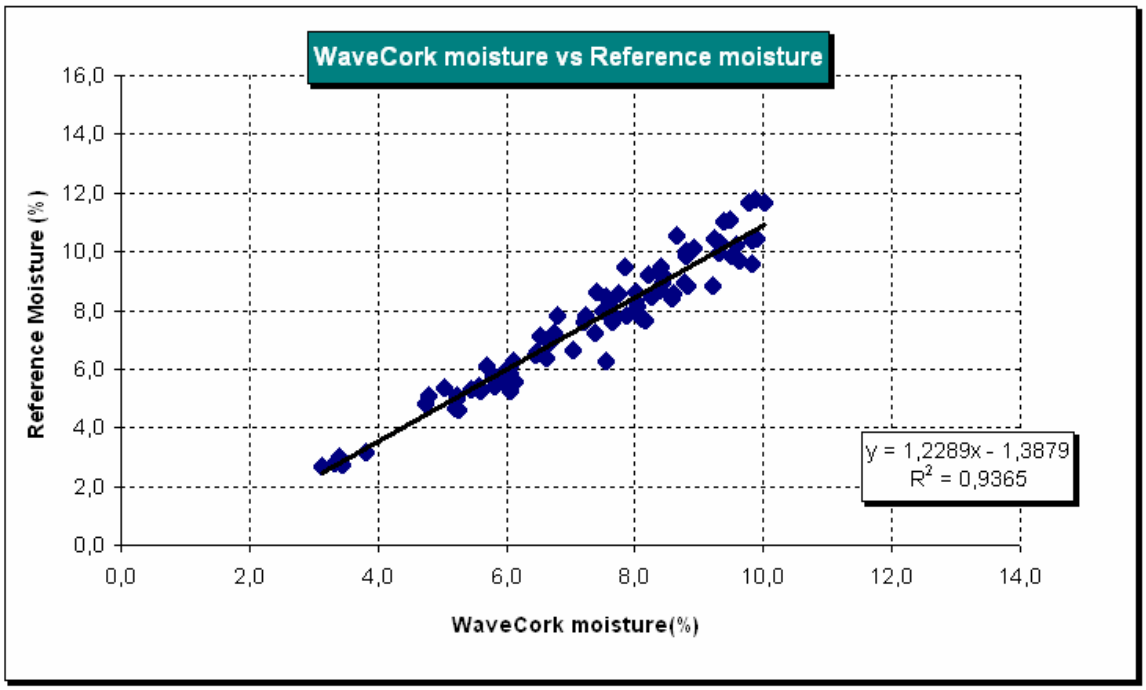


**Figure 1**. **Graphical representation of the individual moisture results obtained by direct readings on the microwave hygrometer *WaveCork* vs. moisture content results obtained by applying the reference method.**
***Note:*** *Tests carried out on natural cork stoppers, washed, of nominal size 45x24 mm.*

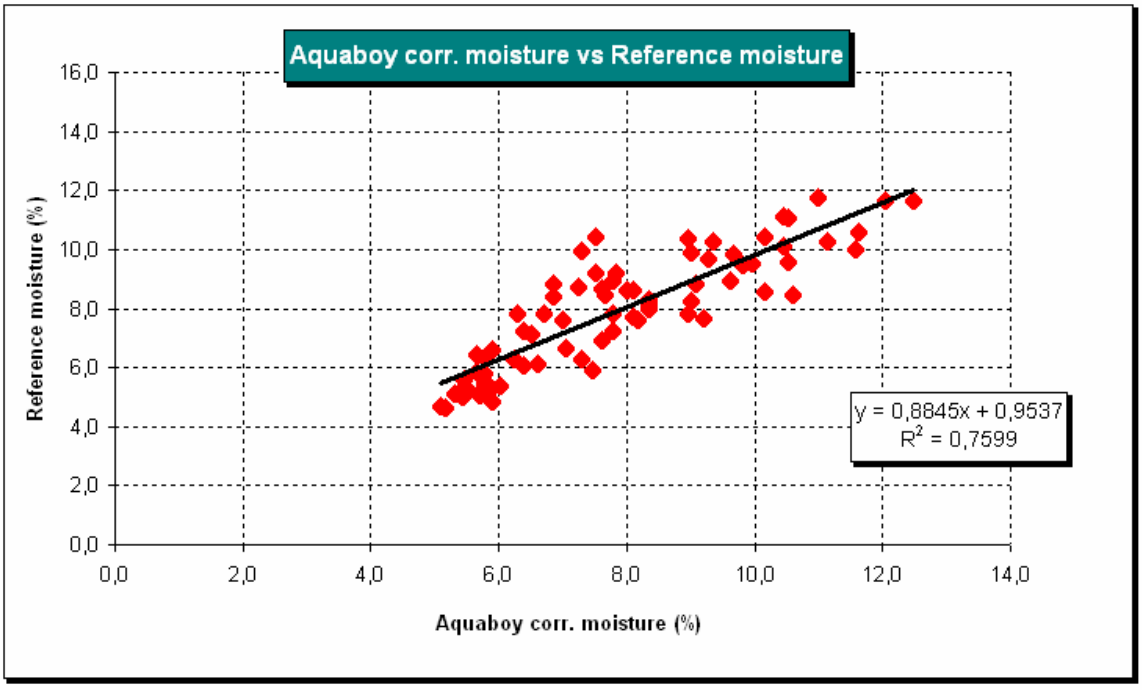


**Figure 2**. **Graphical representation of the individual moisture results obtained by correlated readings on the *Aquaboy* hygrometer vs. moisture content results obtained by applying the reference method.**
***Note:*** *Tests carried out on natural cork stoppers, washed, of nominal size 45x24 mm.*

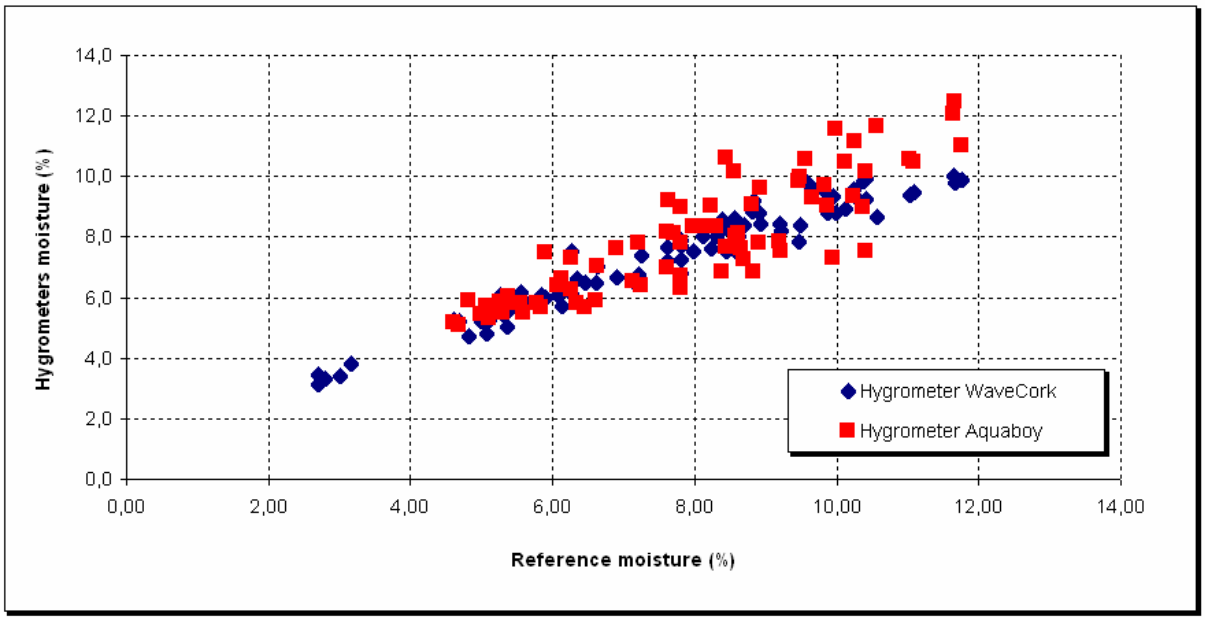


**Figure 3**. **Graphical representation of the individual moisture results obtained by direct readings on the microwave hygrometer *WaveCork* and on the *Aquaboy* hygrometer vs. moisture content results obtained by applying the reference method.**
***Note:*** *Tests carried out on natural cork stoppers, washed, of nominal size 45x24 mm.*

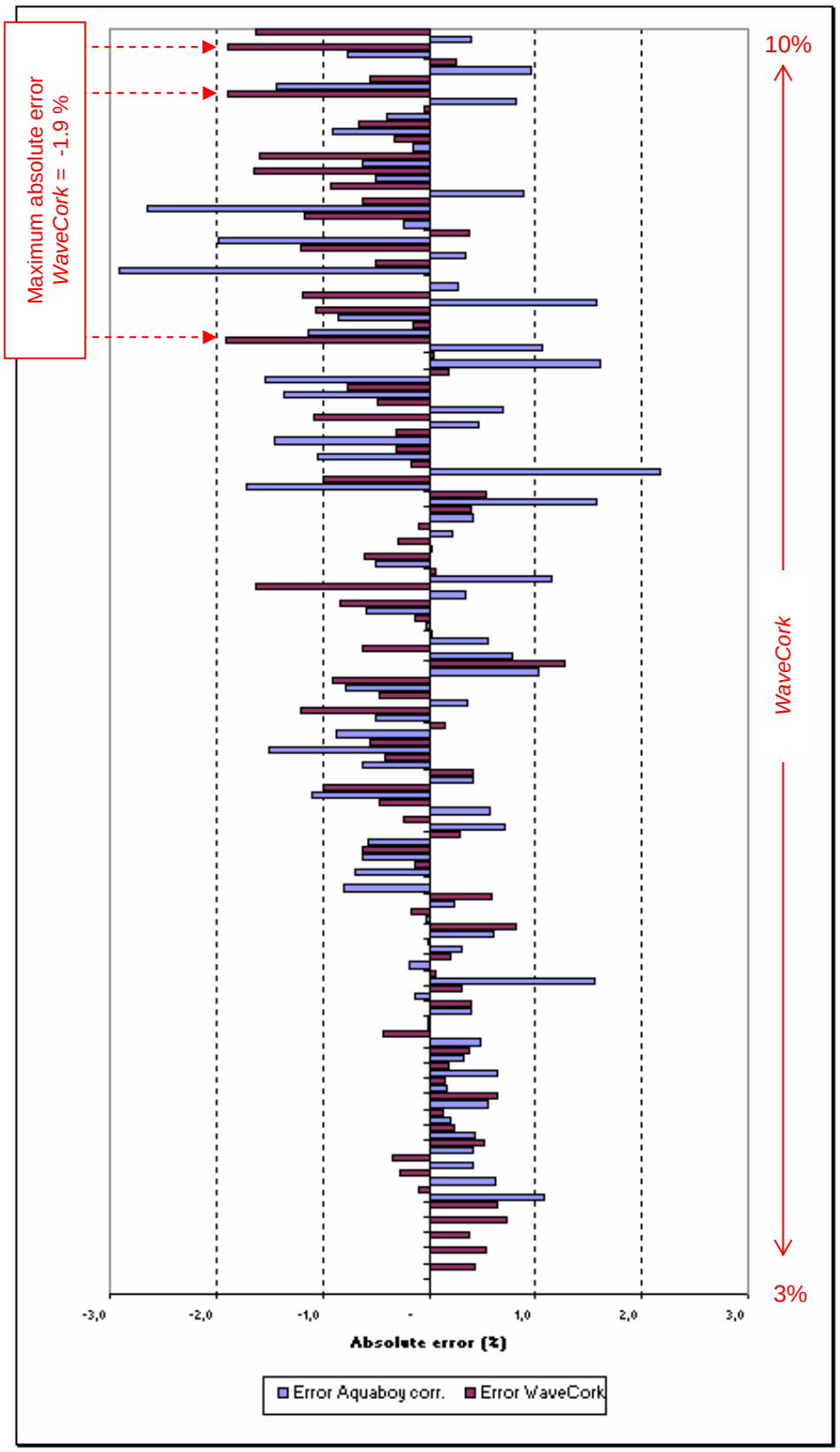


**Figure 4. Graphical representation of the absolute errors distribution obtained for *WaveCork* and *Aquaboy* hygrometers.**

## 7. COMMENTS

Given the experimental approach described above and the analytical results thus determined, it is possible to verify the following:

- The moisture content measurements performed on the present sample of natural cork stoppers, by using the microwave hygrometer *WaveCork*, showed a maximum absolute error of -1.9%.
- The use of the microwave hygrometer *WaveCork* for the determination of the moisture content in natural cork stoppers allowed to obtain values in correlation with the reference method NP ISO 9727-3:2011, by means of the establishment of the following mathematical equation of linear adjustment, guided by a clearly satisfactory correlation coefficient ($R^2$ = 0,936):
  - Reference moisture (%) = 1,229 * *WaveCork* moisture (%) - 1,388
- The trend of linear adjustment evidenced by the microwave hygrometer *WaveCork* ($R^2$ = 0,936) showed a significant qualitative gain, when compared to the characteristic linear adjustement of the *Aquaboy* hygrometer used as an accessory equipment in the realization of the present study ($R^2$ = 0,760).

Santa Maria de Lamas, 18th November 2019

| General Director | Technical Director |
| --- | --- |
| (Alzira Quintanilha) | (Sérgio Moutinho) |