Rapid "moisture" determination as an alternative to CRM76 for modern oral pouches

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INTRODUCTION

The moisture content of modern oral products is a key quality parameter with typical values ranging between 20% and 60%. Both oven mass balance as described in CRM#76, and Karl Fischer determination (ISO6488) are well established. However, a CRM#79 determination will take 3.5hours and is not suitable for process control.

EXPERIMENTAL

Microwave as a potential fast non-destructive method of moisture determination was tested using a TEWS MW4300 fitted with the SLH-50B sensor (TEWS GmbH & Co Ltd, Hamburg, Germany). Using nicotine pouches with known different moisture levels a plot of apparent microwave moisture against true moisture could be formed.

From the graph, as true moisture increases the microwave moisture first increases then decreases giving a flat response in the range 30 to 45% moisture. The variability of individual pouch measurement is poor (high pouch SD) which is exacerbated by the need for exact placement of the pouch in the cavity and the amount of compression of the powder in the pouch.

An alternative system using the surface resistance of a pouch under test was devised and integrated into an Orion test station (Cerulean, Milton Keynes, UK). A small current is passed (<1s) between electrodes and the resistance measured which is calibrated to the moisture of the pouch (assumption: bulk moisture and surface moisture is equivalent at time of test).

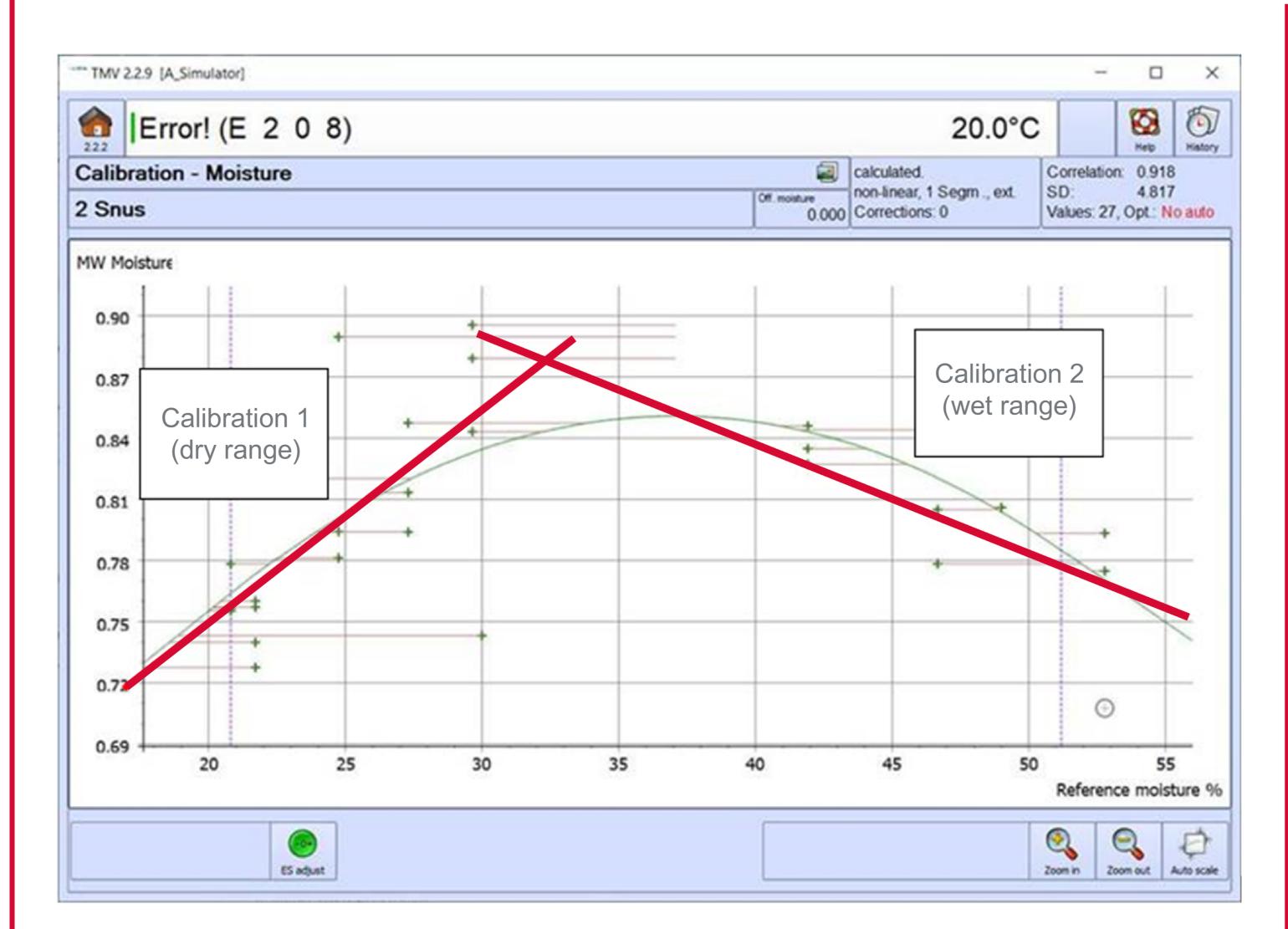


Figure 1: Microwave moisture vs moisture determined by CRM#76. Note similar microwave moistures obtained from two distinctly different "true" moistures.

RESULTS

Four different commercially available brands of pouched products were used for determination. A "generic" calibration curve was derived from repeated measurements of 30+ pouch types whilst brand specific calibration curves were derived using A2D counts from the resistive measurement cross referenced against oven volatile "moisture".

CONCLUSIONS

Above 10% moisture the resistive method is fast, and suitably accurate for process control. The need for a brand specific calibration is trivial.

Microwave measurement could be developed to give a fast wide range measurement device but cannot rival the simplicaity of a resistive system.

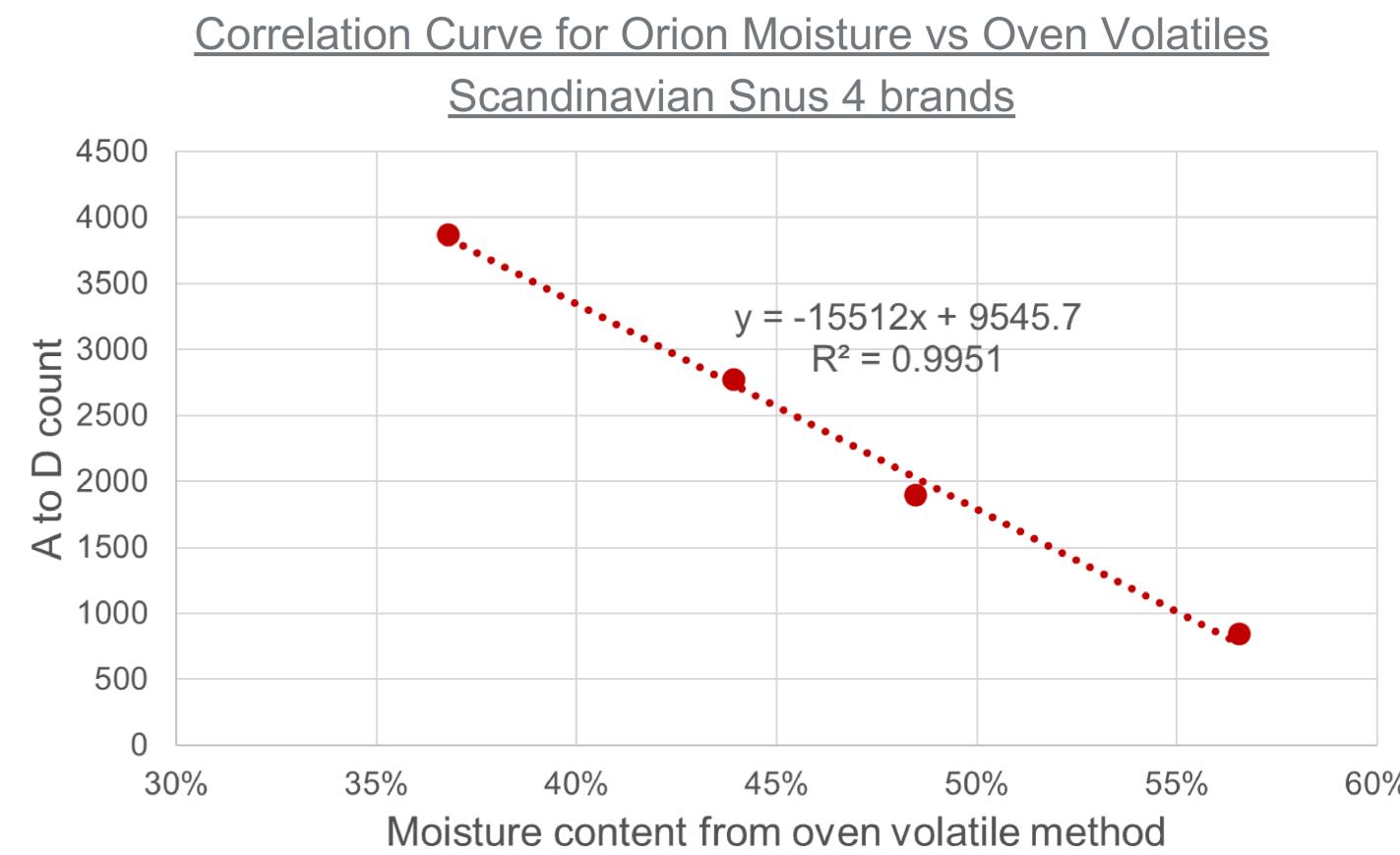


Figure 2: correlation of resistive A2D counts with CRM#76 oven volatile method using generic calibration curve

The figures show for brand specific derived calibration curves the correlation to CRM#76 is good (within 2% difference on average – figure 2) allowing that pouch-to-pouch variability can vary from 1% to 8% moisture.

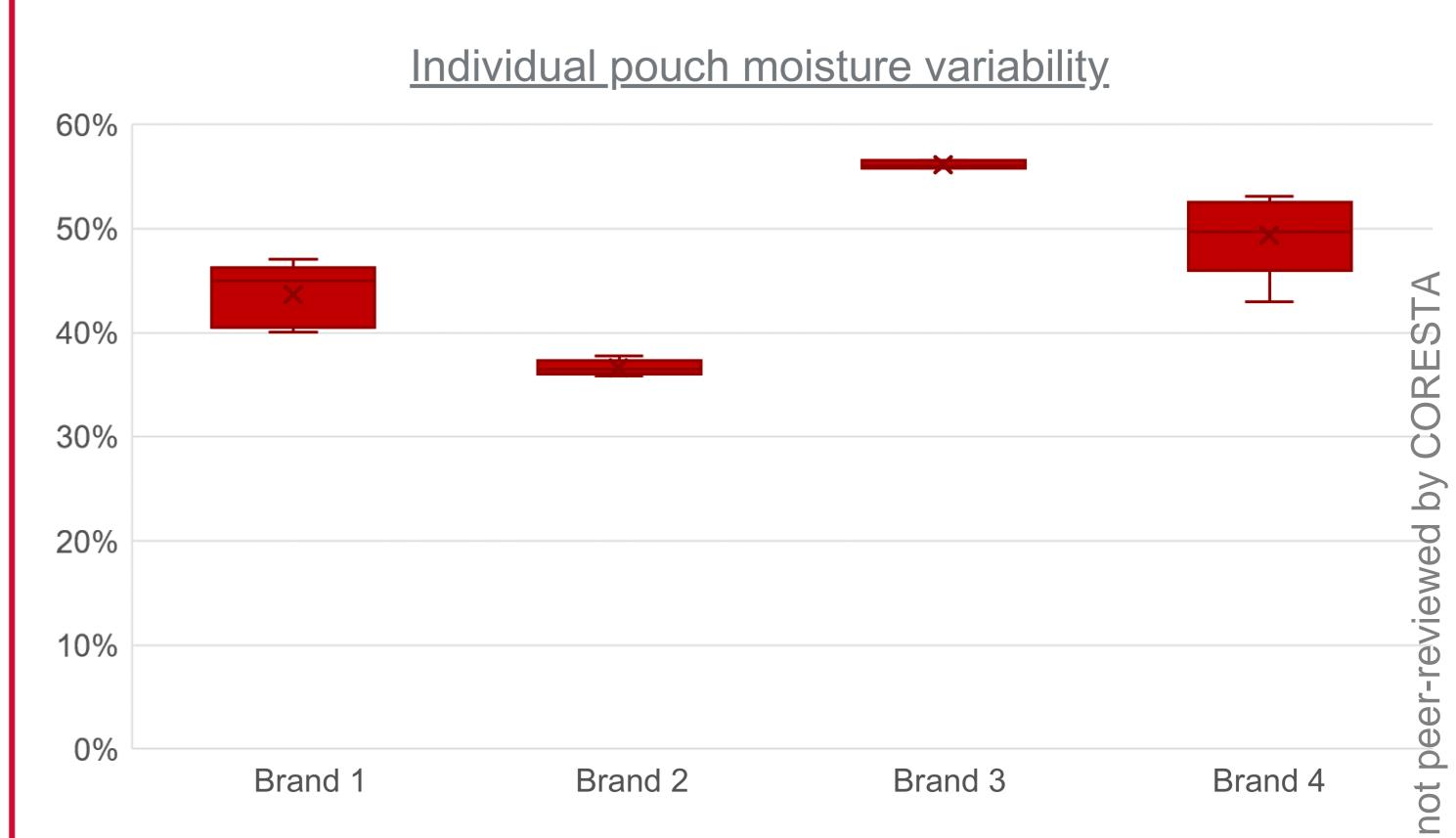


Figure 3: Individual pouch moisture variability for 4 commercially obtained brands. Range of moistures for individual pouches lie between ±1% and ±8%

