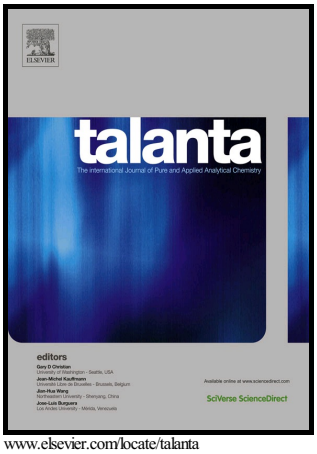

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Near Infra-Red spectroscopy for content uniformity of powder blends– focus on calibration set development, orthogonality transfer and robustness testing

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ABSTRACT:
The aim of this work was to develop and validate a NIR method for the quantification of three active ingredients from powder blends. Calibration set formulations were selected based on a D-optimal experimental design with three factors (ibuprofen, paracetamol, caffeine) and five variation levels (80-90-100-110-120%). NIR spectra were recorded in transmittance mode using a rotating sample configuration. Prior to model development the effect of spectral pre-processing was assessed by evaluating its impact over the transfer of orthogonality from concentration space to spectral space. NIR method was validated on the full calibration range with external prediction sets, using the accuracy profile approach. Robustness testing results showed that the accuracy of predictions for the analyte found in lower concentrations (caffeine) was influenced by relative humidity, while paracetamol/ibuprofen predictions were robust to all factors. Redefinition of interfering factor variation level was beneficial to reduce the bias in caffeine content predictions. Also, alternative solutions are provided for ensuring robustness and successful routine use.