IDENTIFICATION OF FALSIFIED DRUGS USING NEAR-INFRARED SPECTROSCOPY

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Near-Infrared Spectroscopy (NIRS) was investigated aiming at the identification of falsified drugs. The identification is based on comparison of the NIR spectrum of a sample with a typical spectra of an authentic drug using multivariate modelling and classification algorithms (PCA/SIMCA).

Two spectrophotometers (Brimrose - Luminar 2000 and 2030), based on acoustic-optical filter (AOTF) technology, sharing the same controlling computer, software (Brimrose - Snap 2.03) and the data acquisition electronics, were employed. The Luminar 2000 scans the range 850 1800 nm and was employed for transmitance/absorbance measurements of liquids with a transflectance optical bundle probe with total optical path of 5 mm and a circular area of 0.5 cm2. Model 2030 scans the rage 1100 2400 nm and was employed for reflectance measurement of solids drugs. 300 spectra, acquired in about 20 s, were averaged for each sample. Chemometric treatment of the spectral data, modelling and classification were performed by using the Unscrambler 7.5 software (CAMO Norway). This package provides the Principal Component Analysis (PCA) and SIMCA algorithms, used for modelling and classification, respectively.

Initially, NIRS was evaluated for spectrum acquisition of various drugs, selected in order to accomplish the diversity of physico-chemical characteristics found among commercial products. Parameters which could affect the spectra of a given drug (especially if presented as solid tablets) were investigated and the results showed that the first derivative can minimise spectral changes associated with tablet geometry, physical differences in their faces and position in relation to the probe beam. The effect of ambient humidity and temperature were also investigated. The first factor needs to be controlled for model construction because the ambient humidity can cause spectral alterations that should cause the wrong classification of a real drug if the factor is not considered by the model.

A protocol is proposed to construct a multivariate model and to include it in a library enabling testing for drug authenticity. The protocol recommends the inclusion of at least 50 spectra of a given drug presented as tablets. These spectra must be obtained using both faces of 25 samples and collected from different batches found in commerce. PCA is applied and a SIMCA classification is used for drug identification.

The capability of the NIRS in distinguish among drugs with similar composition has been demonstrated by constructing models for Aspirin, Melhoral, AAS and Doril, all nominally containing 500 mg of acetyl salicylic acid as active ingredient and presenting slight differences in composition coming from the inactive ingredients and/or small quantities of accompanying drugs, such as caffeine.

The methodology was evaluated with ten known samples of falsified drugs and was able to recognise all of them as false. The results show unequivocally the potential of NIRS for rapid, on-site, and non-destructive identification of falsified pharmaceuticals.