J. Pharm. Pharmacol. 1998, 50 (Supplement): 43

Transfer of near-infrared spectra of solvents between different instruments

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Near infrared spectroscopy is a technique that is gaining increasing recognition within the pharmaceutical industry and the ability to transfer data between different instruments is now receiving considerable attention from within the industry and regulatory authorities (Moffat et al 1997). It is well known that subtle differences between spectra arising from instrumental variations can affect quantitative determinations (Blank et al 1996). However, the effects on identification processes are less well documented. This work aims to determine if a library of solvents constructed for identification purposes can be successfully transferred between 4 different instrumental systems.

Spectra were measured by transflectance using different Foss NIRSystems (Silverspring, USA) instruments. Each solvent spectrum was the mean of 32 scans and recorded over the range of 1100 to 2200 nm.The instrumental systems examined were:
S1 : 6500 spectrophotometer/Smartprobe,
S2 : 6500 spectrophotometer/Rapid Content Analyzer (a sample cup module),
S3 :6500 spectrophotometer/ Immersion probe,
S4 :5000 spectrophotometer /Smartprobe.
(Note: the same monochromator was used for S1 and S2).
The 13 solvents used were : acetone, acetonitrile, butan-2-ol, butan-1-ol, dichloromethane, N,N-dimethylformamide, absolute alcohol, ethyl acetate, methanol, propan-1-ol, propan-2-ol, toluene and tetrahydrofuran .

Spectra were compared by calculating the correlation coefficient (*r*) in wavelength space. The 52 spectra measured gave rise to 1326 (i.e. $C_{52,2}$) different possible pairs of which 78 are pairs of the same solvents.Using the second derivative (absorbance) spectra, a good separation between correct matches and correct mismatches was observed, Figure 1. Seventy five of the correct matches had *r* values ≥ 0.99. N,N - dimethylformamide as measured on *S1* and *S2* gave an *r* value of only 0.954 and this is believed to be due to water uptake of this hygroscopic solvent between measurements. The highest correlation between spectra of different solvents was 0.93, observed for propan-1-ol and butan-1-ol when recorded using *S1* and *S2* respectively.

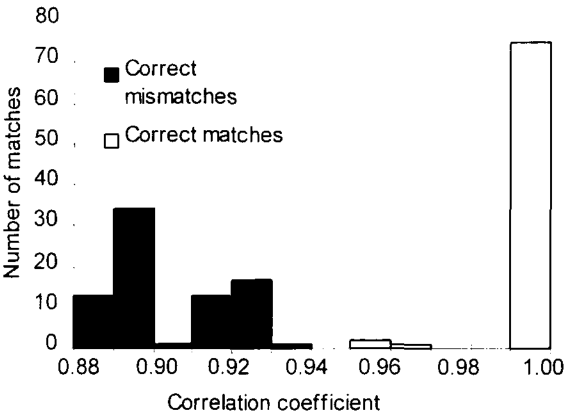

Figure 1 Distribution of correlation cofficient values from a library of 13 solvents using the different instrumental systems

Further validation studies were carried out to investigate the effects of temperature, water and other impurities on the values of *r*. A threshold value of 0.95 was found to give an identification method which was robust and transferable . This work has shown that an NIRS identification application can be successfully transferred across different instrumental setups.

References

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