Transmission Raman Measurements: Experimental Comparison Between HES2000 and Czerny Turner Instrument

Ref: HES-ISI-RP-0010 01

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INTRODUCTION

In recent years spatially offset and transmission Raman measurements have gained popularity in a variety of sectors, including the pharmaceutical and forensics industries. The key advantage of transmission Raman in particular is that it provides a bulk measurement of the sample and is therefore resistant to false returns. A sketch of a typical transmission Raman spectrometer setup is shown in Figure 1. The laser light is fired at the tablet or sample, the light then passes through the tablet exciting Raman events. The Raman photons are observed from a significant portion of the tablet surface area, resulting in the sample source itself having a large étendue. In addition, this signal can be weak for large samples (thickness ≥ 5mm) due to attenuation. In order to capture a significant portion of this light and provide high resolution measurements the spectrometer must have a large étendue. However, in traditional fibre coupled Czerny Turner systems an input slit of 50 µm is required in order to achieve resolutions of < 10 cm^{-1}, restricting the instrument's light gathering potential. One solution is to use fibre bundles that capture the light from multiple points. This, however, can be complex and expensive.

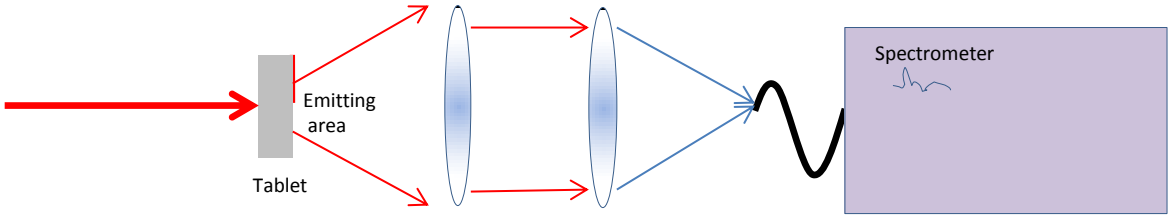

Figure 1 *Typical Transmission Raman Setup*

The HES2000 spectrometer offered by ISI can capture the light from a 1 mm input fibre, requires no slit whilst maintaining a high level of spectral resolution (3 cm^{-1} per measurement bin). In this document we discuss an experimental comparison campaign between a HES2000 instrument and a market-leading fibre coupled Czerny Turner instrument to make Transmission Raman measurements of a 5 mm thick paracetamol tablet.

EXPERIMENTAL SETUP

The paracetamol tablet was illuminated by a fibre coupled 785 nm CW laser. The light passing through was then captured via a 50 mm focal length lens, which collimated the light which was then passed through a 785 nm long pass edge filter provided by Semrock. The light was then focused into an optical fibre via a 50 mm focusing lens. The light was captured via a 50 µm core and a 1 mm core optical fibre, which was then used to couple the light into the Czerny Turner and HES2000 instruments. The spectra for each experiment were captured in 3 and 10 seconds respectively. The HES 2000 raman spectrometer is based on a static Fourier

transform spectrometer design and thus has no moving parts while still proving a throughput advantage. Being a FT based instrument data can be post processed using techniques such as zero filling and phase correction.

TEST RESULTS

To ensure both systems were tested in equivalent scenarios, no post processing was applied to the data gathered to ensure a fair comparison. Figure 2 shows the spectrum gathered by the HES2000 instrument using a 10 second integration time and a 1 mm coupling optical fibre.

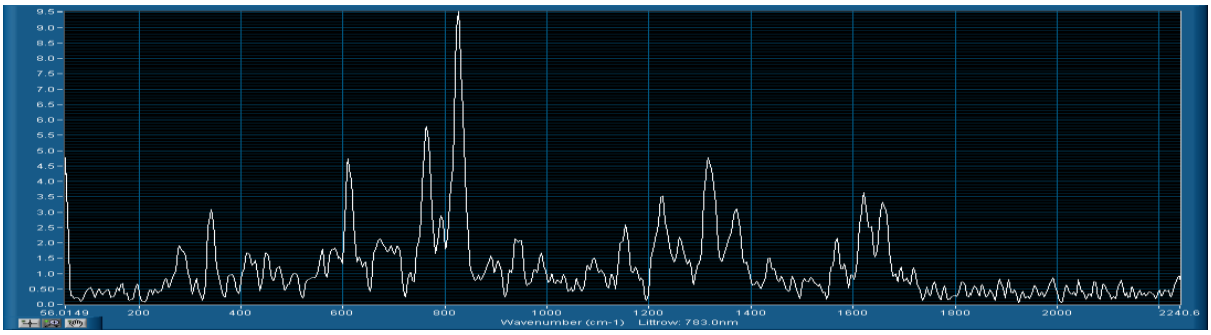

Figure 2 *Paracetamol Raman spectrum acquired with HES200 instrument using 1 mm optical fibre and a 10 second integration time*

The figure demonstrates a clean Raman spectrum from the paracetamol tablet similar to those that can found the literature with at least 13 clear peaks identified. This same fibre was then coupled to the Czerny tuner instrument Figure 3.

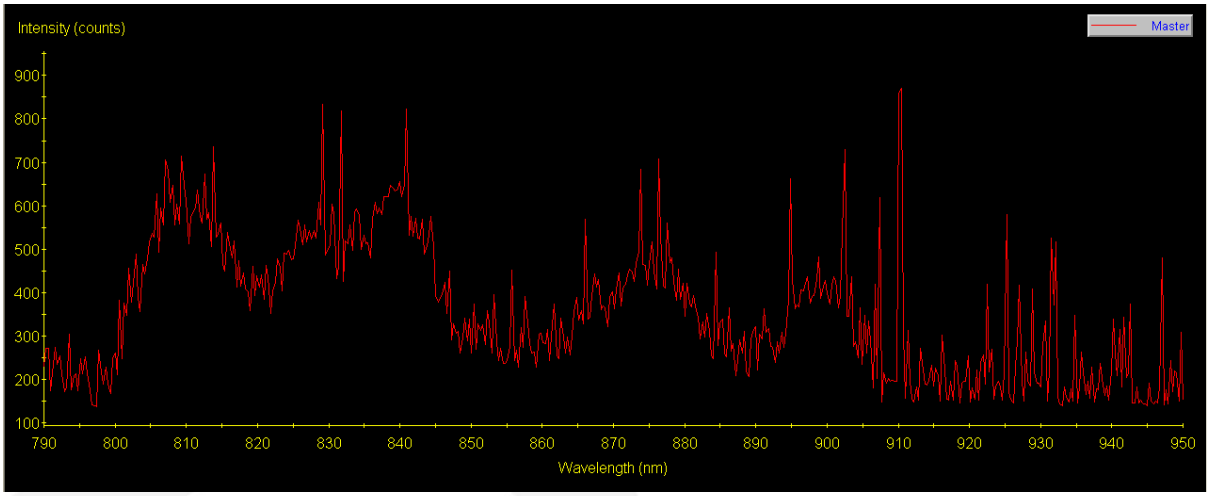

Figure 3 *Czerny Turner spectrum of paracetamol tablet 10 second integration time using 1 mm core optical fibre*

Figure 3 shows that the Czerny Tuner instrument using this fibre does collect the Raman photons, however its resolution is severely affected and thus clean peaks cannot be observed and thus it would not be possible to identify the tablet in a blind test.

Figure 4 shows the Czerny tuner and HES 2000 data normalised and plotted together to illustrate the comparison directly.

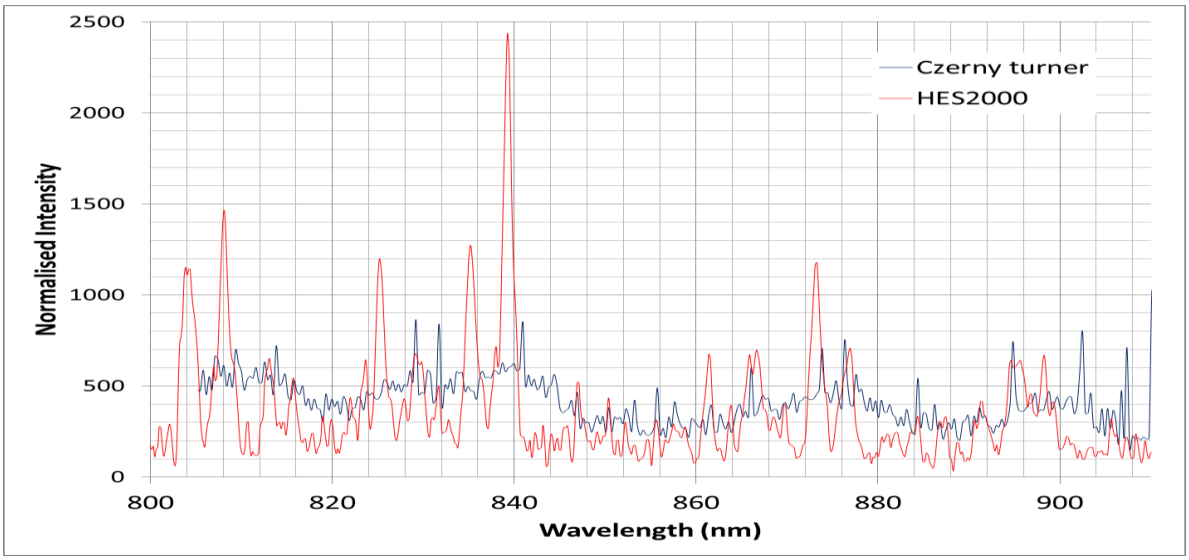

Figure 4 *Transmission Raman measurement through a paracetamol tablet 10 second integration: Blue line is Czerny turner measurement, Red line is the HES 2000 measurement*

The measurements were repeated using a 3 second integration time (Figure 5 and Figure 6)

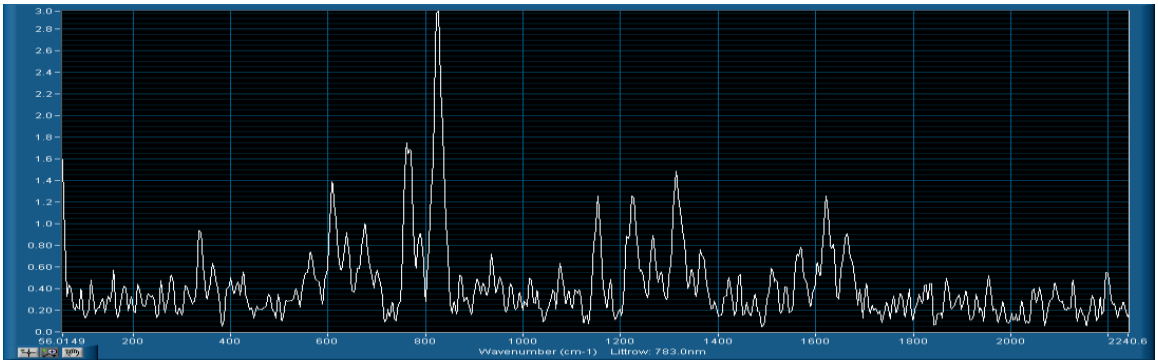

Figure 5 *HES2000 spectrum of paracetamol tablet 3 second integration time using 1 mm core optical fibre*

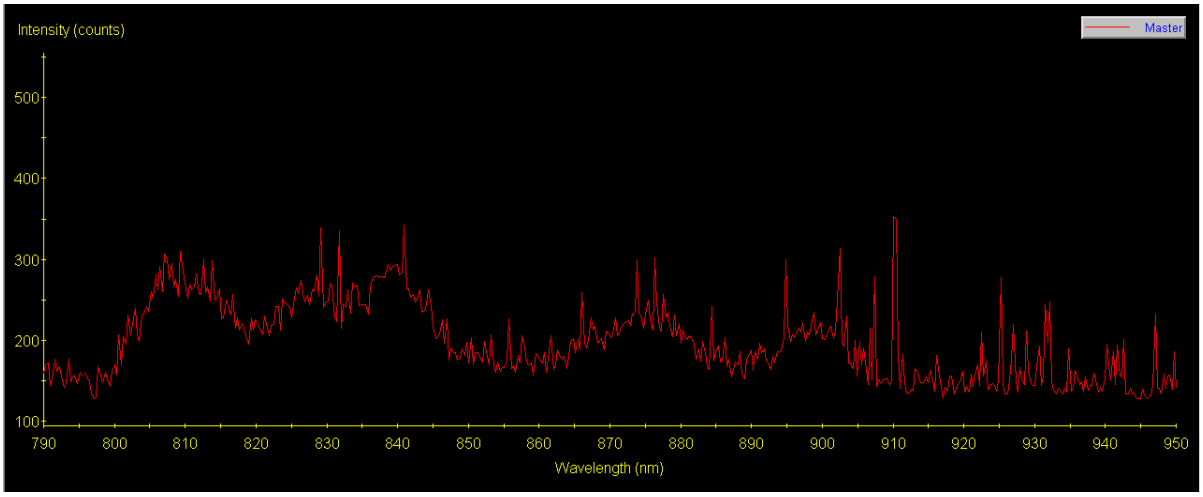

Figure 6 *Czerny Turner spectrum of paracetamol tablet 3 second integration time using 1 mm core optical fibre*

The pattern observed with the 10 second integration measurement is repeated in 3 seconds. The HES2000 instrument observes a clear paracetamol spectrum whereas although the Czerny tuner instrument observes sufficient photons it cannot resolve the spectrum. It should be noted that in the spectra gathered there is still a significant amount of laser light leakage (~ equivalent to 75 % of the total Raman light) . This has no effect of the Czerny tuner instrument but does increase the noise in the HES2000 due to its multiplex nature illustrating that the system would benefit from further filtering.

In order for the Czerny Turner instrument to acquire a spectrum with sufficient resolution to resolve the Raman spectra above, a 50 µm fibre was used to gather the light. However this results in a factor of 400 reduction in the signal, as shown in Figure 7 using a 10 second integration time.

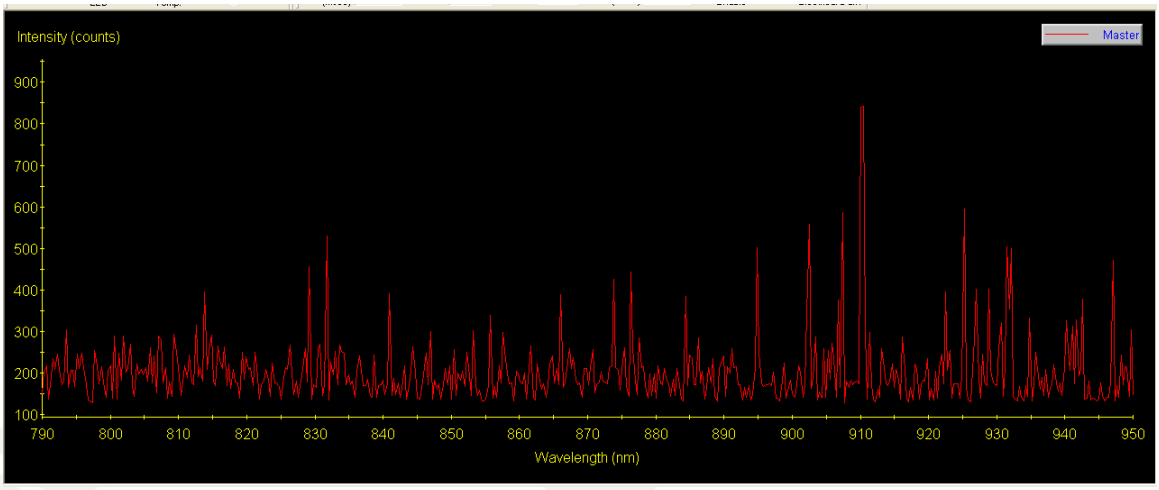

Figure 7 *Czerny Turner spectrum of paracetamol tablet 10 second integration time using 50 µm core optical fibre*

The resolution of the HES 2000 is unaffected by fitting a 50 µm however the signal it gathered is also dramatically reduced and thus no clear spectrum is observed within the 10 second integration time limit.

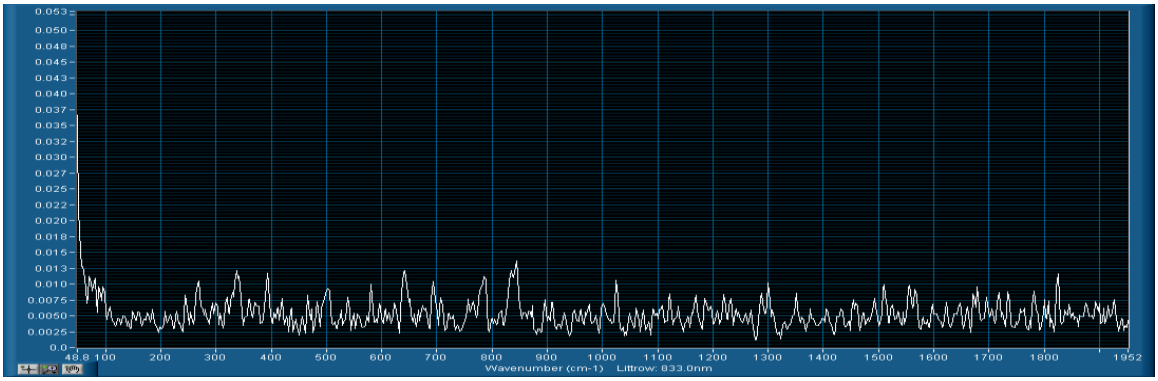

Figure 8 *HES 2000 spectrum of paracetamol tablet using a 50 µm core fibre and a 10 second integration time*

CONCLUSIONS

An experimental comparison has been made between a HES2000 instrument and a fibre coupled Czerny Turner system. Both systems ability to make transmission Raman measurements of a 5 mm thick paracetamol tablet have been examined. In making this type of measurement the étendue of the spectrometer is the limiting factor, as illustrated by the results gathered.

The HES spectrometer observed good quality spectra in a few seconds with high resolution when using a 1 mm aperture optical fibre. The Czerny Turner must sacrifice resolution to gather the light, in which case the sample cannot be clearly identified. In order to provide sufficient resolution the Czerny Tuner instrument requires a 50 µm core fibre. When using such a small fibre neither spectrometer can observe the Raman spectrum in this experimental setup.

The performance of the HES2000 could be improved further by adding an edge filter into the spectrometer to reduce stray laser light and, in addition, post processing techniques such as zero filling and phase correction could be used to improve the contrast of the peaks in the data.

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