Comparing pharmaceuticals as 7 mm & 13 mm KBr Pellets

Both Specac's Basic Solid Pack and Advanced Solid Pack offer unique benefits for FTIR pellet preparation.

IR SPECTROSCOPY IS A WELL ESTABLISHED TECHNIQUE to identify and distinguish samples in the gas, solid and liquid phases.

A sample is typically sandwiched between two windows, separated by a known pathlength. **Potassium bromide** (KBr, spectroscopic grade) is typically used as the window material because it is **transparent in the IR**, between 4000–400 cm^{-1}.

Alternatively, samples can be contained within a KBr matrix and **pressed to form a pellet** that is then analysed.

This paper compares the IR spectra of **7 mm and 13 mm diameter pellets** of three pharmaceutical samples, shown in Table 1.

Sample	Sample Form/ Description	Chemical Formula	Figure
Buscopan	White powder	$C_{21}H_{30}NO_4^+$	1
Levothyroxine	White powder	$C_{15}H_{11}I_4NO_4$	2
Omeprazole	White powder	$C_{17}H_{19}N_3O_3S$	3

Table 1: Chemical formula for the active pharmaceutical sample.

Equipment and Method

Samples were ground in **an agate pestle and mortar**.

A small amount (1 wt%) was mixed with an excess of KBr powder (99 wt%) and ground down again to form a uniform consistency.

Aliquots of the mixture were used to make the 7 mm and 13 mm diameter pellets for each sample.

The IR spectra of both pellet sizes for each sample were recorded using **an FTIR spectrometer at standard room temperature with a resolution of 4 cm^{-1}**.

The Specac **Basic Solid Pack** was used to make the 7 mm diameter KBr sample pellets with a 2T Mini-Pellet press.

The Specac **Advanced Solid Pack**, with its 13 mm Evacuable Pellet Die and a 15T Manual Hydraulic Press, was used to make the 13 mm diameter KBr pellets.

Discussion

The transmission spectra for 7 and 13 mm pellets of **Buscopan, Levothyroxine and Omeprazole** are presented in Figures 1, 2 and 3 respectively.

For each sample, there is excellent agreement with respect to relative peak intensity and position, regardless of the pellet size used.

The only significant difference between the 7 mm and 13 mm pellets is that the absorption band intensities for 13 mm pellet samples are twice as large as those for the 7 mm pellet.

This is simply a consequence of the different **relative concentration** of sample present interacting with the IR light beam over a given surface area.

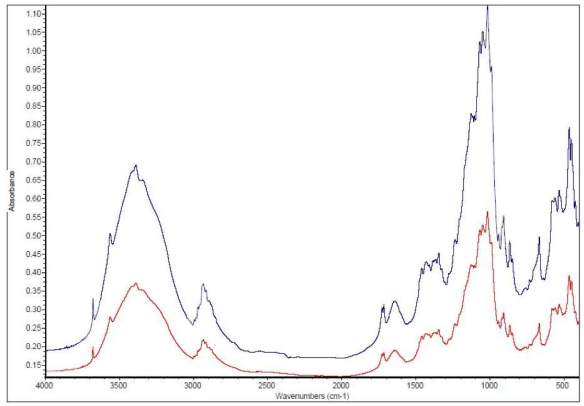

Figure 1: Comparison of FTIR spectra of Buscopan as 7 mm (red) & 13 mm (blue) KBr Pellets.

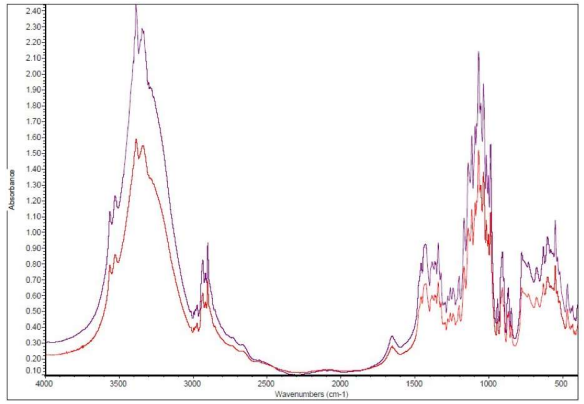

Figure 2: Comparison of FTIR spectra of Levothyroxine as 7 mm (red) & 13 mm (blue) KBr Pellets.

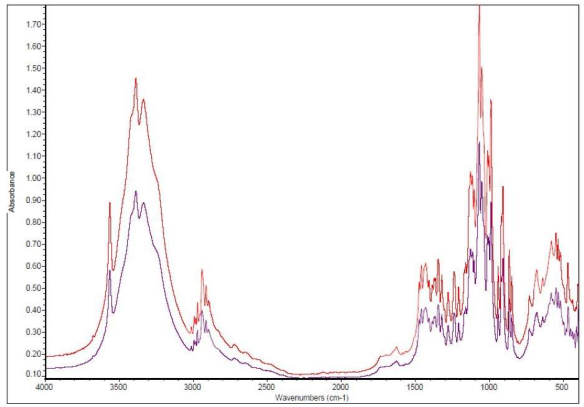

Figure 3: Comparison of FTIR spectra of Omeprazole as 7 mm (blue) & 13 mm (red) KBr Pellets.

@Specac
Specac Ltd

> They consistently produce high quality spectra.

Figure 4 shows the spectrum of the Omeprazole 7 mm pellet, which has been scaled by a factor of ~1.5, overlapped with the spectrum of 13 mm pellet.

There is **a perfect match** for the peaks in the finger print region and only minor differences around longer wavenumber. This reinforces the fact that both sizes of pellets produce identical spectra.

Basic Solid Pack	Part Number
2T Mini-Pellet Press	GS03940
7 mm Pellet Die + Ring Holder	GS03950
Spare Ring Holder	GS03951
Pestle and Mortar	GS03600
50 g KBr Powder	GS03610
7 mm Disc Holder Mount	GS03960

Table 2: The Basic Solid Pack

Advanced Solid Pack	Part Number
15T Manual Hydraulic Press	GS15011
13 mm Disc Holder with Mount	GS03410
Pestle and Mortar	GS03600
50 g KBr Powder	GS03610
13 mm Evacuable Pellet Die	GS03000

Table 3: The Advanced Solid Pack

Conclusion

Three solid pharmaceutical samples were mixed with KBr powder to make 7 mm and 13 mm pellets and analysed using IR spectroscopy.

The different sized pellets were pressed using **a 2 ton and 15 ton press**, respectively. The concentration of the samples in the KBr matrix was sufficient enough to resolve all the spectral bands for both pellets.

For each sample, the IR spectra for both pellet sizes **showed no differences in their qualitative and quantitative interpretations**.

Hence, it is important to compare the equipment used. **A load of ~7 tons is needed to make the 13 mm diameter KBr pellet**, which required the 15T Manual Hydraulic Press.

This press is large, heavy and less mobile. Therefore, **the samples must be brought *to* the press**. Conversely, to make a 7 mm wide KBr pellet a load of ~2 tons is sufficient and the 2T Mini-Pellet Press was used.

This press is small, light and portable, hence **KBr pellet production can be done locally** without the need to move the sample. This is important if the samples are prepared in a particular environment.

The equipment needed to make **7 mm pellets are much cheaper** while the pellets themselves are slightly easier and faster to produce. Moreover, they consistently produce high quality spectra similar to the 13 mm pellets.

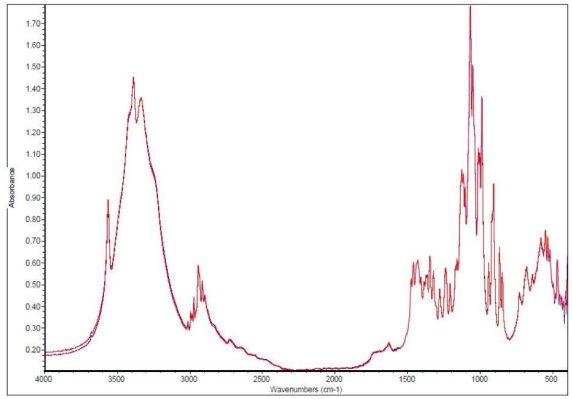

Figure 4: Scaled FTIR spectra of Omeprazole as 7 mm (blue) and 13 mm (red) KBr Pellets.