

FT-NIR Spectroscopy

Creating Custom Tablet Holders for the Tablet Autosampler



Introduction

The Tablet Autosampler System consists of a Spectrum3™ FT-NIR Spectrometer coupled to an autosampler with transmission/ reflectance optics and InGaAs detector.

In order to optimize energy throughput and minimize stray light, the PerkinElmer Custom Mold System was developed.

A previous approach to eliminating stray light used optical aperture diaphragms. These consist of a series of thin blades which can be sized to alter the aperture (Figure 1).

The tablet is then pressed onto the top surface of the aperture. A major problem with this is that the aperture makes a poor light seal with the surface of the tablet.

The aperture is not smooth, but has a series of overlapping blades, allowing stray light to leak through the spaces between the overlapping blades and the tablet. This is a problem even with round, flat tablets. The problem is further exacerbated using lozenge-, triangular- or irregular shaped tablets which do not fit well onto a simple circular aperture.

Achieving a Light-Tight Seal - The Custom Mold

The custom mold system is used to cast an opaque mold around the base of the tablet, which makes a very accurate and light-tight seal. The effect of this is to dramatically reduce stray light. A series of identical molds are created, ensuring consistently high quality data with increased measurement throughput.

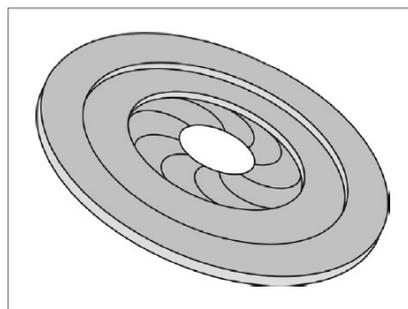


Figure 1. Aperture Diaphragm.

Making the Custom Mold

A sample is adhered to a molding pin using cyanoacrylate adhesive (Figure 2). The tablet is then treated with a release agent to ensure clean release of the mold after formation (Figure 3).

The molding pin, with tablet attached, is then inserted into the mold former (Figure 4). This is half-filled with a filled polyurethane elastomer (Figure 5), which has a working time of around 45 minutes.

The assembly is left to cure overnight at room temperature, then the tablet and pin are removed

(Figure 6). The mold is removed from the mold former (Figure 7), and is ready for use.

The process is repeated to allow automation. The Tablet Autosampler carousel holds up to 30 samples including reference. Tablets for measurement are inserted into the molds (Figure 8), these are then ready for loading (Figure 9) onto the carousel (Figure 10). The mold has a flat side to ensure that it is always placed in the carousel with the same orientation.

The Custom Mold Kit

All parts required for the creation of more than a hundred custom molds are included in the custom mold kit (part number L1245025). Custom molds can be re-used for measurements up to 100 times. Five 6 mm and five 4 mm tablet setting pins are included in the kit, so 10 molds can be routinely produced with the standard kit. Pins can be ordered separately to increase mold formation throughput. Tablet Mold consumables are listed in Table 1.

Note: the resin has a shelf life of six months.

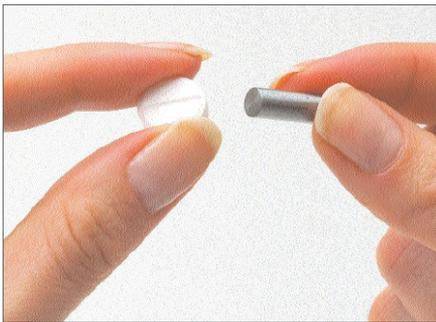


Figure 2. Attaching the tablet to the molding pin.



Figure 3. Treating the tablet with release agent.



Figure 4. Inserting into the mold former.



Figure 5. Adding elastomer.



Figure 6. Removing the molding pin.



Figure 7. Removing the mold.



Figure 8. Inserting a tablet for analysis.



Figure 9. Ready for analysis.



Figure 10. The custom mold system and autosampler carousel.

Verifying Stray Light Rejection

NIR absorbance spectra of a proprietary aspirin (round, approximately 10 mm diameter) tablet and a crescent-shaped tablet (the pale green tablet in Figure 13) were collected using the Custom Mold and an aperture diaphragm. The background was collected before each measurement using the empty custom mold or the empty diaphragm.

Spectra were collected in transmission mode between 12000 cm^{-1} and 6000 cm^{-1} at 16 cm^{-1} resolution. Eight spectra were accumulated.

Table 1. Custom tablet mold resin kit contents and consumables.

AUTOSAMPLER MOULDING KIT (L1245025) CONTAINS:		
Part Number	Component	Units
04947633	No. 6 Large Pencil Brush	1
09200010	Syringe- 1 mL Disposable	100
09200011	Syringe- 10 mL Disposable	100
09200012	Beaker - 100 mL Tri-Corner	100
09200013	Syringe Nozzle Gray	50
09200017	Spatula Nickel/Stainless Steel	3
L1241588	Tablet Setting Pin 6 mm Diameter	5
L1241592	Tablet Setting Pin 4 mm Diameter	5
L1241590	Tablet Mold for 6 mm Pin	5
L1241591	Tablet Mold for 4 mm Pin	5
L1241589	Tablet Mold Base	5
L1245024	Custom Tablet Molds Resin Kit	1

CUSTOM TABLET MOLDS RESIN KIT (L1245024) CONTAINS:		
Part Number	Component	Units
04940236	Cyanoacrylate Adhesive	5 mL
09200016	Resin Kit (Resin and Accelerator)	500 g
L1245124	Release Agent	30 mL

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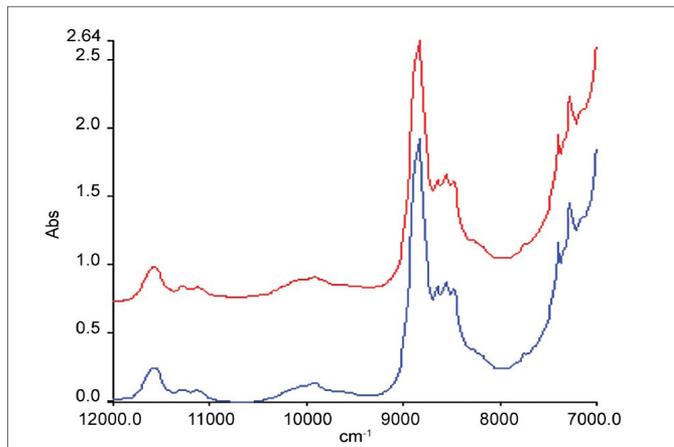


Figure 11. Comparison of stray light using a diaphragm aperture (blue) and the custom mold (red) for a round tablet.

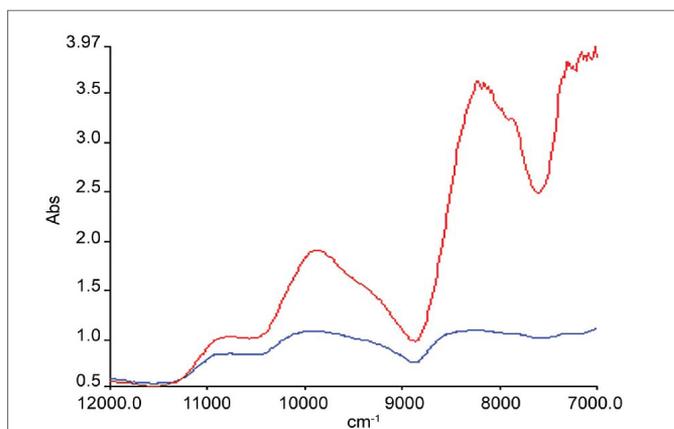


Figure 12. Comparison of stray light using a diaphragm aperture (blue) and the custom mold (red) for a crescent-shaped tablet.

Results

Figure 11 shows the absorbance spectra for a round aspirin tablet using a custom mold prepared for that tablet and an aperture iris. Stray light in the apertured tablet caused a large-scale change in baseline compared to the custom mold system. Figure 12 shows a similar comparison for the pale green crescent-shaped tablet in Figure 10. In this case, not only is there a large baseline shift, but also large scale distortion of the absorbance spectrum.

Conclusion

The custom mold system optimizes data quality by making an extremely close seal between the tablet and the surrounding material in the mold. This minimizes stray light providing higher dynamic range and superior quality spectra.

