

COMBINING MICRO X-RAY FLUORESCENCE AND INFRARED IMAGING  
SPECTROSCOPIES FOR THE UNDERSTANDING OF MODEL AND  
COMMERCIAL PHARMACEUTICAL TABLET SYSTEMS.

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Micro X-ray Fluorescence (MXRF) is a potent elemental technique that provides both single point spectra and full spectral elemental maps in a 10 x 10 cm area. The X-ray spot size of a commercial instrument is approximately 50 micrometers; therefore, a map may contain between 1600 and 40,000 discrete spectra. MXRF images are important because they provide several orders of information, including quantitative and qualitative information of elemental species as well as heterogeneities and spatial distribution of the species present.

Infrared (IR) microscopic imaging provides molecular information that compliments MXRF. The Perkin-Elmer Spotlight 300 provides a high signal-to-noise single point detector, as well as an innovative 16 x 1 pixel array for faster mapping than the single pixel can provide. Typical IR beam spot size in imaging mode is 25 or 6.25 micrometers. By using this array, images up to 5 x 5 cm can be generated. Image size is not limited by the size of the camera as in a focal plane array system.

Generating both MXRF and IR full spectral maps of a sample provides a synergistic layer of information that improves the understanding of the sample's composition. Collecting full elemental and molecular spectra at each and every point in the sample makes it possible to import both image cubes into a chemometric software package, overlay the elemental and molecular images, and draw cross correlations. All of this makes it possible to extract unexpected information well after data collection. Issues to be addressed include: sample registration, instrument conditions, and technique characteristics such as beam penetration depth and how each affects post processing. A model pharmaceutical tablet consisting of a hydroxyethylcellulose binder, calcium phosphate filler, magnesium stearate lubricant and acetaminophen as the active ingredient, and commercial vitamin B12 tablets will be presented that demonstrate the power of integrating these spectroscopic imaging techniques.

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