FT-NIR Tests for GMP Compliance

by Cynthia Kradjel and John Richmond

By June 2010, 16 years after the Dietary Supplement Health and Education Act of 1994 (DSHEA) became law, all dietary supplement manufacturers were required to implement and be in full compliance with the requirements of the dietary supplement cGMP (current good manufacturing practice) regulations [21 CFR 111].

In addition to the requirements that help ensure consistent identity, purity, strength and composition, the regulations (111.75 [a][i]) specifically require dietary supplement manufacturers to “conduct at least one appropriate test or examination to verify the identity of any component that is a dietary ingredient.” And, “You must ensure that the tests and examinations that you use to determine whether the specifications are met are appropriate, scientifically valid methods” (111.75[h][i]). In its audits, FDA is finding some dietary supplement manufacturers do not have suitable identity methods in place.

Given that the dietary supplement industry uses natural products that are complex materials and that raw material/ingredient inventories may contain hundreds of ingredients that constantly change because of new developments in the field or the demands of the marketplace, it is no wonder this minimum requirement to verify the identity of dietary ingredients has been a daunting endeavor in terms of manpower, time, effort and cost.

To help comply with these regulations, commercially available Fourier transform near infrared analysis (FT-NIR) technology provides a scientifically valid, easy-to-use, fit-for-purpose, validated and fast solution for identity testing; “pass/fail” results can be available within minutes. And, most importantly, the technology is accepted by FDA. “We went through a full FDA inspection [recently]. They [FDA] spent a whole day going through the ... equipment [NIR instrumentation] and reviewed our standard operating procedures (SOPs) and analysis work, and we passed the inspection without problems,” said Mark Glazier, president, Nutrilite division of Amway.

Although NIR has been long used for the quantitative and qualitative analyses of a diverse range of materials including pharmaceuticals, fit-for-purpose NIR systems suitable for regulatory purposes for the analysis of highly complex natural product-based dietary ingredients used in the dietary supplements industry are a more recent development. Companies have had to modify existing software for NIR systems to meet the requirements of 21 CFR Part 11 (electronic records and electronic signatures).

Industry collaboration is also helping manufacturers to meet the testing standards. For example, ChromaDex and Bruker Optics developed the Comply ID program, a type of “plug-and-play” identity program. The equipment is installed at the manufacturer’s facility; samples are scanned onsite and the data analyzed remotely at an offsite location where an extensive ingredient database is stored for identity testing. After the test is complete, a pass/fail report is sent back to the customer. ChromaDex and Bruker are collectively building an ingredients database in a cloud system that will, in essence, develop into the sum total of the data from all participants in the program.

Company-specific and customized libraries work well, too. At NutraBio, dietary ingredients qualified by a third party in triplicate are used to build the database. “For qualification purposes, analysis by a third party is not costing [more] because we are doing it anyway as part of our vendor qualification,” Glazier said. “We might have four manufacturers for one ingredient, so for each of those manufacturers we do three sets of qualifications that [give] 12 [lots] that have been proven out and that [we use for] building our library (database). And, the more samples we test and add to the library, the more robust it becomes.”

In addition to the required regulatory identity testing, chemists at Dynamic Pharmaceuticals Inc. are also building custom libraries for the quick identification of in-process blends, “So that we can quickly [verify] the identity and the content uniformity of our blends and know they’re ready for manufacturing,” explained Chris Reckner, president, Dynamic Pharmaceuticals. Reckner also noted that based on previous experience with NIR systems, “It is very important to establish, which we did with Bruker, criteria for the purchase of the instrumentation as well as expectations in terms of training, technical support and troubleshooting.”

From the standpoint of the speed of analysis, NIR is fast; samples can be analyzed in seconds, either with specially designed fiber optic probes or in the lab. In fact, compliance with the regulations of foreign markets is important for companies that export finished products. “In one particular foreign market, the requirement to verify the identity of each container of dietary ingredient provided the justification for buying the NIR [analyzer] in the first place,” said Mark Dan, quality control chemist, Nutrilite division of Amway.

Cynthia Kradjel is business development manager, and John Richmond is the vice president, NIR and process technology, at Bruker Optics Inc. (BrukerOptics.com).

Reprinted with permission from Natural Products Insider, October 2011. © 2011 VIRGO Publishing. All Rights Reserved.