

Arkworld Manufacturers Quality Assurance

By Jeff Golini

American consumers are increasingly supplementing or replacing prescription medicines with medicinal herbs, vitamins and minerals. An estimated \$ 12 billion dollars was spent by Americans on natural supplements in 1997. In addition, the consumption of functional foods, nutraceutical drinks, and other types of dietary supplements are growing at a rapid pace.

Analytical requirements for this industry are characterized by the following key issues:

1. The physical state of incoming raw materials, in-process formulations and final products can span a diverse range. For example, herbs can be formulated as capsules, tinctures, extracts, teas or added to common foods such as fruit drinks or potato chips. Samples may be liquids in the form of sublingual, tinctures, homeopathic, vitamins, cough and cold medicines, or special drinks; powders in the form of weight gain formulas, protein, carbohydrate or baby powders, cocoa mixes, drink mixes or spices; or capsules of vitamins, minerals, and herbs that may be clear, colored or printed on the exterior.
2. Natural products have complex matrixes, which may contribute to their benefit for the consumer. The ability to measure the product without separating the constituents from their matrix is very important.
3. Botanicals and phytochemicals and other supplements are regulated by the 1994 Dietary Supplement Health and Education Act. Industry leaders are striving to set high quality standards and implement Good manufacturing practice (GMP) principles in their manufacturing facilities.
4. Speed and volume requirements make a fast testing method essential. For example, a shipment of incoming raw material may arrive in 25 drums, depending upon the volume. With traditional laboratory methods, it would be very time consuming to verify the identity and quality of every container of incoming raw material. A method that could be used in the receiving area and produce results in less than one minute would make the task achievable.
5. For companies that sell standardized herbals, there is an increasing challenge to develop analytical methods to accurately quantify the assay. Potency of herbs can vary from batch to batch so in order to use the NF (National Formulary) designation on the label, standardization testing must be performed.
6. Purity testing is becoming more important as demand for botanicals, herbals and natural products grow, supply issues put pressure on raw material suppliers.

Manufacturers must look for the presence of spiked or doped material. Similar raw materials can often fool traditional tests, so manufacturers order a \$2000/kg raw material and may receive a \$200/kg similar raw material.

7. A new federal rule published September 23, 1997 dictates changes to labeling and includes a box of “supplement facts” similar to the “nutrition facts” label found on food products. Standardized products will state the amount of active ingredient in each dose. Herbal products will be identified by the common or usual name the labels and include which part of the plant is used, such as the root, stem or leaf. These new requirements challenge traditional testing methods.
8. Products tend to move quickly into and out of the marketplace. Method development must be flexible, rapid and responsive.

An analytical technology has been identified and implemented to address the eight key issues effectively. This technology is Near Infrared Analysis. Heavily used in the pharmaceutical industry, the technology is based the interaction of light with the material to be analyzed. Sophisticated mathematical algorithms are used to store, interpret and present results.

Near Infrared Technology has been successfully implemented at Arkworld using the Bran + Luebbe InfraProver for routine testing of identity and quality of all inbound raw materials as ingredients for health foods and nutritionally beneficial formulations.

Every container entering the facility is tested to guarantee that identity, purity, safety, and potency standards are met. The containers are sampled and then quarantined until completely approved. Once testing is completed and the material is approved, each ingredient is issued a Certificate of Analysis which corresponds to each Lot number. Their products range from a wide variety of health food ingredients, herbals and nutritional supplements. The products range in physical form from solids, liquids, gels and tablets. Qualitative methods are used for identity and quality testing and quantitative methods are used for in-process optimization and for generation data for Certificate of Analysis (COA).

The Bran + Luebbe InfraProver is a polarization interferometer Fourier transform NIR instrument based on principles of HPCS (High Performance Crystal Spectroscopy). The system uses a moving crystal wedge to obtain FT Interferograms for the materials being tested; resulting in a substantial reduction in sensitivity to vibration compared to traditional lab grating and FT instruments. This type of optical system enables the instrument to be placed on a cart and moved to the receiving area or warehouse area, without compromising the precision of the results.

The design of the instrument allows All American Pharmaceutical to present a wide variety of sample forms to the system via software control without changing the hardware. Two options are available for solids.

A 2-meter fiber optic probe enables “probing” of containers in the reflectance mode. Materials can be scanned directly in the containers or through plastic packaging material. Alternatively, the Solid Presentation Accessory (SPA) permits spectroscopic interrogation through the base of a vial when the vial is placed on the optical window of the SPA. Tablets or capsules can also be analyzed with this accessory. Liquids were scanned in the transmission cell, a “built-in” cuvette chamber located in the center of the InfraProver.

Long term stability of the instrument is achieved via internal laser referencing for all wavelengths ensuring wavelength accuracy and reproducibility.

Near Infrared technology scans the product in its entirety. Mathematical algorithms then separate the chemical or physical properties of interest from the rest of the matrix. Samples are therefore analyzed “as –is “ with no sample preparation

The InfraProver was originally developed for the pharmaceutical industry so it was designed to meet GLP, GMP principles. Many pharmaceutical companies have validated the instrument for their applications.

Once a calibration has been developed, identity, quality and quantity can be analyzed in less than 60 seconds. Operator level functions are present for routine use.

Assay methods have been developed enabling All American Pharmaceutical to meet new label requirements. Purity can be determined, giving manufacturing the information that incoming raw materials have been spiked or doped. In some cases, NIR has shown that similar materials were substituted or co-mingled with incoming raw materials.

Method development can be turned around to adapt the technology to new products or new formulations.