Instrumental Methods for Determination of Oil, Protein and Other Constituents of Oilseeds and Meals

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The need to assess quality at all points in the process and handling system has increased the need for rapid and accurate instrumental methods which can be applied in non-laboratory settings. The Oil Protein Symposium at the 1993 AOCS Annual Meeting highlighted developments in three areas, near-infrared reflectance (NIR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy and combustion-based nitrogen analysis. Over the past 25 years two instrumental methods, NIR and NMR, have developed to the point where they have become important tools in the rapid assessment of oilseed and meal quality, both in process and handling systems, and as routine tools in plant breeding programs and in agronomy and quality monitoring studies. More recently, the development of combustion-based nitrogen analyzers capable of automatic analysis and utilizing relatively large sample sizes have given oilseed analysts a more environmentally friendly alternative to the Kjeldahl method.

NIR was discussed in three papers ("Near-Infrared Spectroscopic Analysis of Deterioration Indices of Soybeans for Process Control in Milling Plant," by T. Sato, H. Abe, S. Kawano, G. Veno, K. Suzuki and M. Iwamoto; "Comparison of Three Whole Seed Near-Infrared Analyzers for Measuring Quality Components of Canola Seed," by J.K. Daun, K.M. Clear and P. Williams; and "Identification and Segregation of High-Value Soybeans at a Country Elevator," by Charles R. Hurburgh, Jr.); and NMR in one paper ("Simultaneous Determination of Oil and Water Contents in Different Oilseeds by Pulsed Nuclear Magnetic Resonance," by G. Rubel) which described the pulsed NMR method. The combustion method for nitrogen determination was described in the third paper ("Comparison of Combustion and Kjeldahl Methods for Determination of Nitrogen in Oilseeds," by J.K. Daun and D.G. DeClercq). These papers all show actual experimental data evaluating the analytical techniques or demonstrating the use of the methods in different situations.

Hurburgh demonstrated that whole seed NIR analysis can allow successful and profitable segregation of soybeans by protein level at a country elevator. Sato *et al.* demonstrated the use of NIR to determine several factors, including major components (oil, protein and moisture) and some important minor components (nitrogen solubility index, germination, acid value and digestibility). Daun *et al.* compared three different whole seed analyzers for the determination of different quality characteristics in canola and showed that in order to determine all the important quality characteristics it was necessary to measure the spectrum from 600 nm (chlorophyll) through to 2500 nm (glucosinolates). In this way they determined fatty acid composition (saturated fatty acids, oleic acid, linoleic acid, linolenic acid).

Rubel presented a paper on the use of pulsed NMR to determine the oil and moisture content of different oilseeds. Continuous-wave NMR has been used for routine determination of oil content in component pricing, plant breeding and quality surveys for many years and is the subject of reference methods from both ISO and FOSFA. Pulsed NMR has an advantage in that the sample need not be dried prior to analysis (although the moisture content must be less than 10%). This allows the sample to be analyzed quickly and, in the case of plant breeding programs, does not destroy the viability of the seeds. An ISO standard for pulsed NMR determination of oil and protein has been published.

Two papers on determination of total nitrogen in oilseeds by combustion were also presented. One [Biscak, R., *J. Assoc. Off. Anal. Chem. 76*:780 (1993)] described the results of a collaborative study comparing the combustion method with Kjeldahl. This paper provided the basis for the adoption of the combustion method by AOAC International. Daun *et al.* compared the combustion method to the Kjeldahl procedure used by the Canadian Grain Commission. The combustion method gave higher results than Kjeldahl for canola, flaxseed, soybeans and mustard seed. Results for sunflowerseed were more variable, possibly due to difficulties in grinding the seed.

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