

for domestic (USA) consumption, state the amount of total fat and saturated fat in the product. For the past several years NIST has developed and refined nutritional standard reference materials (SRMs), based on food matrices, to provide reliable measurements of nutritionally significant analytes to the food testing community. SRMs can also be used for quality assurance when assigning values to in-house control materials. NIST has recently developed three food based SRM materials with contrasting total fat content and fatty acid profiles. SRM 1946, Lake Superior Fish Tissue is a frozen fish tissue homogenate, prepared from freshwater trout (*Salvelinus namaycush*) collected from Lake Superior. It is a relatively low (~10%) fat, high protein material, with a high proportion of mono- and poly-unsaturated fats. A unit of SRM 1946 consists of four 10 g jars (wet basis) of cryogenically homogenized fillet tissue homogenate. SRM 2384, Baking Chocolate is a high (over 50 percent) fat, high carbohydrate material. A few saturated and mono-unsaturated materials dominate the fatty acid profile of SRM 2384. One unit of SRM 2384 consists of five 91 g bars of chocolate. Twelve fatty acids are certified in SRM 2387 Peanut Butter. Five major components make up almost 50% of the mass of the material. They are palmitic (C16:0), stearic (C18:0), oleic (C18:1), linoleic (C18:2), and docosanoic (C22:0) acids. A unit of SRM 2387 consists of three 170 g jars of peanut butter. The NIST Certificates of Analysis provide up to three categories of assigned values, i.e., certified, reference, and information values. The majority of fatty acid mass fractions are reported as certified values. Certified values for these materials are based on combining NIST data with data from interlaboratory comparison exercises of the National Food Processors Association (NFPA). Values are reported on an as-received (not dry-mass) basis in mass fraction units.

A Rapid and Routine Method for Determination of Total Glucosinolates in Rapeseed. H. Safafar and M. Kazemzadeh, ORDC, Iran.

Glucose was liberated from glucosinolates using endogenous enzyme after crushing the seed and making a paste with water. Same procedure for the determination of free glucose content of seed was done with acidified methanol. Extracts were mixed with fine charcoal and filtered through Whatman No.1 filter paper to remove interfering phenolic materials. Glucose was measured by a glucose oxidase/peroxidase colorimetric method. Results shown good agreements with other glucose assay methods and with official HPLC method.

Comparison between Kjeldahl and Dumas Methods For Protein Determination In Soy Products. S. Jung^{1,2}, D. Ricker¹, N. Deak¹, E. Aldin¹, J. Recknor³, L. Johnson^{1,2} and P.M. Murphy^{1,2,1} CCUR, USA, ²Dept. of Food Science and Human Nutrition, USA, ³Depart. of Statistics and Statistical Laboratory, USA.

Recently, combustion-type nitrogen analyzers, based on the Dumas method, have emerged presenting the advantages of semi-automatic operation, rapid analysis, and freedom from corrosive and hazardous chemicals resulting in a safe procedure, as an alternative to the well-known Kjeldahl analysis. However, based on literature data, it appeared that the nitrogen content determined by the Dumas method resulted in higher nitrogen values than the Kjeldahl method for some products and there are very limited data available for soy and soy products. The purpose of this study was to determine the relationship between the Dumas and Kjeldahl methods for soy products. Nine soy products were chosen to cover protein content (nitrogen content*6.25) from 0.5% to 90% (wt./wt.). Two Kjeldahl methods (AOAC method 960.52 and the Corn Refiners Association Macro-Kjeldahl method A-18) were used. A Nitrogen Analyzer (Rapid N III) from Elementar Americas, Inc. (Mt Laurel, NJ) was used for the Dumas method. The same sample materials were analyzed on two consecutive days to determine day-to-day variability. For both methods the result for each sample was the mean of four determinations. The data were evaluated by using SAS 8.2, a statistical package of the SAS Institute, Inc. (Cary, NC). There was a

significant difference ($p < 0.05$) between the two Kjeldahl methods used. This difference was correlated to an analyst effect. There was no day-to-day variability with the nitrogen analyzer, between 1.5 to 90% protein content. However, the variability became significant if our lowest concentration (0.5%) was included. There was not significant evidence of a difference in the standard errors for the nitrogen analyzer and the Kjeldahl method. The comparison between the averages of the Kjeldahl and Dumas methods revealed that the nitrogen analyzer gave significantly higher values. The mean ratio between Kjeldahl and Dumas (K/D) varied between 1.03 and 0.94 except for the lowest protein concentrations (0.657). The equation proposed to predict the Kjeldahl measurements is: $y = -0.00536 + 0.97188x$, where y represents the Kjeldahl protein % and x the Dumas protein %. The combustion method for protein determination performed very satisfactorily and could replace the Kjeldahl method for soy and soy products, with the use of a correction factor.

Oxidation of Conjugated Linoleic Acid Methyl Ester. P. Delmonte¹, T. Vogel¹, J.A.G Roach¹, J.K.G Kramer² and M.P. Yurawecz^{1*}, ¹U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, College Park, MD, USA, ²Agric & Agri-Food Canada, Food Research Center, Canada.

The oxidation of CLA FAME isomers is compared with other common FAME at room temperature in the presence and absence of ambient light using glass and polypropylene vials. The reaction proceeds substantially faster in glass than in polypropylene. After 8 days of oxidation with ambient air and laboratory light, oxidation of CLA was faster than linoleate, but slower than that of linolenate. Each of the CLA FAME isomers examined (9c,11t; 9c,11c; 9t,11t; 10t,12c) had a unique oxidation profile. Oxygen addition to the carbon-carbon double bonds in 1,2, 1,3, and 1,4 mechanisms are discussed. Light and oxygen alone were not efficient initiators of oxidation on the polypropylene surface.

Silver Ion and Reverse Phase HPLC of CLA Fatty Acid Methyl Esters. P. Delmonte and M.P. Yurawecz, U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, USA.

Silver ion high performance liquid chromatography is routinely used in the separation of conjugated linoleic acid methyl esters. Using 3 Ag+HPLC columns in series, with a mobile phase of 0.1% acetonitrile/0.5% ethyl ether/hexane it is possible to separate most of the CLA commonly found in food. The use of relative retention times to toluene and 9c,11t-18:2 simplifies the interpretation of chromatograms. This is necessary to compensate for the drift in retention times due to the low solubility of acetonitrile in hexane. The use of 2% acetic acid/hexane as a mobile phase under the same set of conditions provides a similar elution pattern with little loss of resolution, but the stability and reproducibility of retention times simplifies the use of Ag+ HPLC in the purification of compounds. Furthermore, the 10t,12c and 10c,12t isomers, which elute in a single peak using a MeCN/ethyl ether/hexane mobile phase, resolve partially under these conditions. The same set of conditions may be applied at the analysis of CLA as free fatty acids, providing a similar pattern of elution, but longer retention times. In reverse phase chromatography, CLA-FAME shows a stronger interaction with the stationary phase when the elution temperature is reduced below to their melting points. Using a silica based RP18 at 0 or 5 degree Celsius with a 100% acetonitrile mobile phase at 1ml/min, it is possible to achieve a complete baseline resolution of the 9c,11t and 10t,12c isomers in less than 10 minutes. Furthermore, low temperature reverse phase chromatography separates cis and trans monoenes from c/t-CLA, eliminating the cis-monoenes interferences with the c/t-CLA that occurs in silver ion chromatography. Lowering the temperature results in longer retention times and the peak to peak resolution increases, but at the same time, peak tailing increases. Under these conditions, the chromatography of all the geometric isomers from 6,8-18:2 to 13,15-18:2 has been obtained at different temperatures.

Measurable Effects of Microwave Heating on Vegetable Shortenings. E.S.