XVII 1-03  
**OXYGEN BARRIER PROPERTIES OF WHEY PROTEIN ISOLATE COATINGS ON POLYPROPYLENE FILMS**
S.-I. Hong and J.M. Krochta

Oxygen permeation characteristics of whey protein isolate (WPI) coatings on polypropylene (PP) films were investigated to examine the feasibility of WPI coating as a novel biopolymer oxygen barrier for food packaging applications. Heat-denatured aqueous solutions of WPI with several plasticizers including glycerol, sorbitol, sucrose, propylene glycol, and polyethylene glycol were applied on the surfaces of PP films previously treated with corona discharge. Among plasticizers used, sucrose conferred the best oxygen barrier property to the WPI-coated films. Oxygen permeability (OP) of the resulting WPI-coated films increased significantly with temperature, showing very good agreement with the Arrhenius model. OP of the coated films also increased exponentially with relative humidity.

XVII 2-03  
**INHIBITION OF LISTERIA MONOCYTOGENES ON HOT DOGS USING ANTIMICROBIAL WHEY PROTEIN-BASED EDIBLE CASINGS**
A. Cagri, Z. Ustunol, W. Osburn, and E.T. Ryser

Whey protein isolate (WPI) films (pH 5.2) containing p-aminobenzoic acid (PABA) were heat-sealed to form casings. Hot dogs prepared with WPI, collagen, or natural casings were cooked, surface-inoculated to contain $10^3$ *Listeria monocytogenes* CFU/g, and examined for numbers of *L. monocytogenes*, mesophilic aerobic bacteria (MAB), lactic acid bacteria (LAB), and yeast/mold during 42 d of storage at 4°C. *Listeria* populations on hot dogs prepared with WPI-1.0%-PABA casings remained relatively unchanged; however, numbers of *Listeria* on hot dogs prepared with WPI-0.0%-PABA, collagen, and natural casings increased about 2.5 logs during 42 d of refrigerated storage. Populations of MAB, LAB, and mold on WPI-1.0%-PABA casings were 1 to 3 logs lower compared to other casings.

XVII 3-03  
**PLASTICIZER EFFECT ON GREASE BARRIER AND COLOR PROPERTIES OF WHEY-PROTEIN COATINGS ON PAPERBOARD**
S.-Y. Lin and J.M. Krochta

Whey protein concentrates with ~80% protein (WPC-80) plasticized with 0.64 $M$ glycerol or sucrose, or with 0.34 or 0.64 $M$ sorbitol or polyethylene glycol (PEG) 200, produced
WPC-80 with hydrolyzed lactose required addition of less sucrose to produce flexible films. WPC-80 films formed as coatings on paperboard gave a grease barrier comparable to WPI film-coatings. Long-term ambient storage of WPC-80 coated paperboard indicated that the use of sucrose as plasticizer imparted good grease resistance and minimized plasticizer migration.

**XVII 4-03 WHEY PROTEIN COATINGS FOR FRESH FRUITS AND RELATIVE HUMIDITY EFFECTS**  
L. Cisneros-Zevallos and J.M. Krochta  

Whey protein isolate (WPI) films have proven to be excellent gas barriers in previous studies, making them potential coatings for fresh produce. WPI-coated apples and controls were stored at 20°C under RH’s ranging from 54 to 92%. Results showed performance of WPI coatings depended on the environment RH. The internal oxygen was lowered, and carbon dioxide increased with decreasing RH conditions. RH did not affect control fruits. At low RH (about 70 to 80% RH), anaerobic respiration was induced in coated fruits due to low oxygen levels (about 0.025 atm). Controlling thickness and film permeability will allow attainment of the appropriate oxygen and carbon dioxide levels for coated fruit.

**XVII 5-03 A PROCESS FOR INCREASING THE FREE FAT CONTENT OF SPRAY-DRIED WHOLE MILK POWDER**  
A.B. Koc, P.H. Heinemann, and G.R. Ziegler  

Exposing spray-dried whole milk powder to high shear and elevated temperature in a twin-screw continuous mixer increased the free fat content. The effects of operating conditions (powder feed rate, processor screw speed, and process temperature) on lactose crystallinity, particle size distribution, color, and moisture content of spray-dried whole milk powder were investigated using response surface methodology. Exposure to elevated temperatures and high shear: (a) increased the free fat to more than 80%, (b) crystallized the lactose, (c) reduced the average volume-based particle size, and (d) broadened the particle size distribution. The raw dry whole milk with creamy-white color turned into an oily paste with bright-yellow color. Processing enhanced the functional properties of spray-dried whole milk powder for milk chocolate manufacture.

**XVII 6-03 STABILITY OF SULFONAMIDES, NITROFURANS, AND CHLORAMPHENICOL RESIDUES IN PRESERVED RAW MILK SAMPLES MEASURED BY LIQUID CHROMATOGRAPHY**  

A stability study was made of 10 antimicrobials: 6 sulfonamides, 3 nitrofurans, and chloramphenicol residues in raw milk samples preserved with 0.1% potassium dichromate (K₂Cr₂O₇) and 0.05% mercuric bichloride (HgCl₂) during cold storage for 7 days. Preserved milk
samples fortified with 50 ppb of each antimicrobial were analyzed by liquid chromatography. Drugs were extracted with chloroform-acetone after solvent evaporation residues were dissolved with aqueous sodium acetate buffer solution (0.02M, pH 4.8), and fat was removed with hexane. Sulfonamides and chloramphenicol were detected at 275 nm (UV) by using a gradient system of sodium acetate buffer solution-acetonitrile starting at 95 + 5 (v/v) and finishing at 80 + 20 (v/v). Nitrofurans were detected at 375 nm (UV) isocratically with sodium acetate buffer solution-acetonitrile (80 + 20, v/v). Residue stability was measured through recovery data. Sulfamethoxazole, sulfachloropyridazine, nitrofurazone, furazolidone, and furaltadone residues remained stable in the presence of either preservative for 7 days. Sulfamethazine and chloramphenicol were not affected by K₂Cr₂O₇, but had significant losses (p <0.05) when HgCl₂ was used, 26.2 and 13.4%, respectively. Average recoveries of sulfamonomethoxine, sulfamerazine, and sulfathiazole significantly decreased by day 7, with losses of 17.1, 17.2, and 23.2% for K₂Cr₂O₇ and 23.3, 20.7, and 48.0% for HgCl₂, respectively. During 5 days of cold storage, all antimicrobials tested, except sulfathiazole, remained stable in milk samples preserved with 0.1% K₂Cr₂O₇ or 0.05% HgCl₂.

**XVII 7-03**

**INTERLABORATORY STUDY OF A MULTIRESIDUE GAS CHROMATOGRAPHIC METHOD FOR DETERMINATION OF ORGANOCHLORINE AND PYRETHROID PESTICIDES AND POLYCHLOROBIPHENYLS IN MILK, FISH, EGGS, AND BEEF FAT**

F. Bordet, D. Inthavong, and J. M. Fremy


An interlaboratory study was conducted to validate a gas chromatographic (GC) method for determination of 21 organochlorine pesticides, 6 pyrethroid pesticides, and 7 polychlorobiphenyl (PCB) congeners in milk, beef fat, fish, and eggs. The method was performed at low contamination levels, which represent relevant contents in food. It enlarges the applicable scope of the reference method to pyrethroid pesticides and proposes the use of solid-phase extraction (SPE) as a cleanup procedure. Cryogenic extraction was made, and SPE cleanup was performed with 2 successive SPE cartridges. After injection of the purified extract onto a GC column, residues were measured by electron capture detection. Food samples (liquid milk, beef fat, mixed fish, and mixed eggs) were prepared, tested for homogeneity, and sent to 17 laboratories in France. Test portions were spiked with 27 pesticides and 7 PCBs at levels from 26 to 45 ng/g into milk. Based on results for spiked samples, the relative standard deviation for repeatability ranged from 1.5 to 6.8% in milk. The relative standard deviation for reproducibility ranged from 33 to 50% in milk. This method showed acceptable intra- and interlaboratory precision data.

**XVII 8-03**

**MODEL STUDIES ON THE DETECTABILITY OF GENETICALLY MODIFIED FEEDS IN MILK**

R.E. Poms, W. Hochsteiner, K. Luger, J. Glossl, and H. Foissy

The aim of this work was to investigate whether a DNA transfer from foodstuffs like soya and maize was analytically detectable in cow’s milk after digestion and transportation via the bloodstream of dairy cows and, thus, whether milk could report for the employment of transgene feeds. Blood, milk, urine, and feces of dairy cows were examined, and foreign DNA was detected by polymerase chain reaction by specifically amplifying a 226-bp fragment of the maize invertase gene and a 118-bp fragment of the soya lectin gene. An intravenous application of purified plant DNA showed a fast elimination of marker DNA in blood. With feeding experiments, it could be demonstrated that a specific DNA transfer from feeds into milk was not detectable. Therefore, foreign DNA in milk cannot serve as an indicator for the employment of transgene feeds unless milk is directly contaminated with feed components or airborne feed particles.

**NEW AND REVISED 3-A STANDARDS**

3-A Sanitary Standards for Sanitary Fittings, Number 63-03. Effective November 24, 2002.


**XVII 9-03 OPTIMIZATION AND VALIDATION OF A RAPID METHOD TO DETERMINE CITRATE AND INORGANIC PHOSPHATE IN MILK BY CAPILLARY ELECTROPHORESIS**

J.M. Izco, M. Tormo, A. Harris, P.S. Tong, and R. Jimenez-Flores


Quantification of phosphate and citrate compounds is very important because their distribution between soluble and colloidal phases of milk and their interactions with milk proteins influence the stability and some functional properties of dairy products. The aim of this work was to optimize and validate a capillary electrophoresis method for the rapid determination of these compounds in milk. Various parameters affecting analysis have been optimized, including type, composition, and pH of the electrolyte, and sample extraction. Ethanol, acetonitrile, sulfuric acid, water at 50°C or at room temperature were tested as sample buffers (SB). Water at room temperature yielded the best overall results and was chosen for further validation. The extraction time was checked and could be shortened to less than 1 min. Also, sample preparation was simplified to pipet 12 µl of milk into 1 ml of water containing 20 ppm of tartaric acid as an internal standard. The linearity of the method was excellent ($R^2 > 0.999$) with CV values of response factors <3%. The detection limits for phosphate and citrate were 5.1 and 2.4 nM, respectively. The accuracy of the method was calculated for each compound (103.2 and 100.3%). In addition, citrate and phosphate content of several commercial milk samples were analyzed by this method, and the results deviated less than 5% from values obtained when analyzing the samples by official methods. To study the versatility of the technique, other dairy products such as cream cheese,
yogurt, or Cheddar cheese were analyzed and accuracy was similar to milk in all products tested. The procedure is rapid and offers a very fast and simple sample preparation. Once the sample has arrived at the laboratory, less than 5 min (including handling, preparation, running, integration, and quantification) are necessary to determine the concentration of citric acid and inorganic phosphate. Because of the speed and accuracy of this method, it is promising as an analytical quantitative testing technique.