[PD4-9] [04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3]]

The preparation and properties of the liquid membrane electrodes for mefenamic acid

NAM SJ*, KIM KY , JUNG MM, KIM DB, HUR MH, AHN MK°

Pusan regional Food & Drug Administration, Kyungsung Univ.

A liquid membrane electrodes for mefenamic acid, flufenamic acid and niflumic acid were investigated. The electroactive substances of the electrode consists of ternary complex of fenmates and Fe-dipyridyl.
The sensing matrix membrane was plasticized with one of 2-nitrophenyloctyl ether, benzyl 2-nitrophenyl ether, 2-fluorophenyl 2-nitrophenyl ether and 2-nitrodiphenyl ether.
The membrane, plasticized with 2-nitrophenyl ether, exhibited a fast, stable and linear response for 10-5~10-3 mol dm-3 mefenamic acid, flufenamic acid and niflumic acid with an anionic slope of 54 ± 0.5 mV, 55 ± 0.5 mV and 41 ± 0.5 mV / concentration decade respectively.
The electrode exhibited food selectivity with respect to inorganic ions and aromatic and aliphatic anions. These sensors were successfully applied to the determination of fenmates in pure form and pharmaceuticals.

[PD4-10] [04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3]]

Properties and Quantitative Analysis of chrysin 7-O-methacrylate

Mi Kyoung Lee, Ji Meyn Jang, Kyoung Soon Kim, Kyoung-Hwan Kang

College of Pharmacy, Seoul National University, Seoul 152-742, Korea, 1Chong Kun Dang Pharmaceutical Corp., Chung nam 330-830, Korea

The derivative of chrysin 7-O-methacrylate was synthesized by condensing methacrylic acid with chrysin in organic solvent, and its structure was identified by NMR, MS, UV, IR etc. We also investigated the physico-chemical properties and set up the quantitative analytical method of this compound. The correlation coefficient of calibration curve on this compound was approximately 0.9999 by absorption spectrophotometry.

[PD4-11] [04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3]]

Studies on Analysis and Stability of Retinol and Retinylpalmitate in Cosmetics

Kim PS°, Choi SS, Kim IK, Yun MO, Son KH, Lee JP, Kim CK*, Jang SJ

Division of Cosmetics and Hygienic product, Department of Drug Evaluation, KFDA, *Chungang University

The reproducible and precise method for quantitative determination of retinol and retinylpalmitate in cosmetics was studied by high performance liquid chromatography, and the stability test was studied. The analysis was performed with NovaPak C18 column, 1.0 Aufs and 325nm. As a mobile phase, 90% methanol was used for retinol and 100% methanol for retinylpalmitate. A linearity for retinol and retinylpalmitate was obtained within the concentration range of 20~100 IU/ml . The

results of recovery test were 96.9~102.4% for retinol and 98.1~104.3% for retinylpalmitate. We also investigated on stability of retinol and retinylpalmitate products after opening according to variation of temperature. The contents of retinol and retinylpalmitate products were decreased in the range of 78.4~95.1% and 40.4~85.7% at cold storage condition, 60.2~85.6% and 20.2~79.7% at room temperature, 12.5~35.2% and 10.0~39.3% at acceleration condition(40℃) after five months. Therefore it is better to store at cold temperature in use.

[PD4-12] [04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3]]

Development of the Diagnostic Method for Galactosemia

Kim MK°, Yoon HR*, Hong SP

College of Pharmacy, Kyung Hee University, *Dept. of Special Biochemistry, Seoul Clinical Laboratories

A new reversed-phase HPLC method has been developed for the determination of galactose in blood spot (50μL) on Guthrie filter paper using 8-Amino-2-naphthalenesulfonic acid (8,2-ANS) as derivatization reagent. In a metabolic pathway from galactose to glucose-6-phosphate, there are three enzymes involved such as galactokinase, transferase, and epimerase. The deficiency of one of these enzymes causes accumulation of galactose in blood, which provides a pathgnomonic marker. Galactose was extracted from blood spot on filter paper and derivatized with 8,2-ANS to produce Schiff bases, and reduced under sodium cyanoborohydride. This is ready for the HPLC analysis for a diagnosis of Galactosemia. The linear range was between 80 nmol and 20 pmol and the limit of detection (S/N=3) of this method was 0.9 ng/mL. The mean recovery of galactose was 104.29 % with a SD of 3.62 %. with correlation coefficiency of 0.9999. The control range of galactose in blood of Korean newborn (specimen collected with 7 days after birth) were below 6mg/dL for male and female (n=5 for each gender) without any difference. We applied 11 anonymous blood spots in which diagnosis has already made by enzyme assay as one of the galactokinase, transferase, or epimerase deficiencies. All the patients blood spots showed abnormal elevation of galactose. These results suggest that the new method is a valuable tool for the diagnosis of metabolic disorder, Galctosemia.

[PD4-13] [04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3]]

Rapid Monitoring of PEGylation Process by Matrix-Assisted Laser Desorption/Ionization Time-of-Flight Mass Spectrometry

Na DH°, Yoon YS, Park MO, Lee KC

Drug Targeting Laboratory, College of Pharmacy, SungKyunKwan University

The covalent attachment of polyethylene glycol (PEG) has received the increasing attention as a well-established technique that has the capacity to overcome several problems of protein and peptide for therapeutic needs. One of the great challenges of PEG chemistry is characterization of PEGylated conjugates. Matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF MS) was investigated as a method for the rapid control and optimization for PEGylation of peptide and protein. RC160 and ricin A chain (RTA) were used as the model systems of peptide and protein, respectively. MALDI-TOF MS was useful for not only determining the true molecular mass of PEGylated species, but also identifying the individual species contained in a preparation. The PEG-RC160s were characterized by a bell shaped distribution of equally spaced molecular ions 44 Da apart, while PEG-RTA having higher molecular weight showed a unresolved peak of the individual oxyethylene unit. MALDI-TOF MS was demonstrated to be also useful for optimizing the PEGylation conditions through the control of the pH and stoichiometry of the components in the reaction. Moreover, it provides the merits of speed, high resolution, small sample requirements, ease of determination, and simple data manipulations over other analytical tools.