[PD4-4] [ 04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3] ]

## Photostability testing of IY-81149, a new Proton Pump Inhibitor

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The study of the degradation of new drug substances under light is an integral part of stress testing because the photodegradation products might appear in the formulation during storage. These actinometric studies are used to identify precautionary measures and specify storage conditions needed in manufacturing or in formulation of the product. Also, these investigations provide valuable information for submission in applications for marketing authorizations for new active substances. Photostability testing of IY-81149, a new PPI substance, exposed to two artificial daylight lamps which have both UV and visible outputs (Dulux EL) and only visible light (Halogen Haloline), under the influence of container types such as amber, clear glass and quartz was studied. Both lamps provided overall illumination of not less than 1.2 million lux hours. We examined for any changes in color of IY-81149 substance and for assay and degradants arose from photochemical degradation processes. The result shows that the order of photoprotection of its container types were amber, quartz and clear glass. IY-81149 substance in amber was found to be photostable because it had no changes in color and was within justified limits ranged not more than 0.1% for each degradants and not more than 1% for total amount of degradants by the ICH stability and impurity guidelines. But in case of clear glass, this substance was photounstable manifesting that its contents decreased less than 99% after 3 weeks with above exposure to both light sources. We concluded the best storage container for IY-81149 was the amber in order to protect from photodegradation if the storage period was more than 3 weeks.

[PD4-5] [ 04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3] ]

## A Study on Pretreatment Method of Grains for Pesticide Residues Analysis (1)

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This study was carried out to investigate optimal pretreatment condition for the pesticide residues in grains, including rice, barley, sticky rice etc. We bought 4 kinds of pesticide free grains and made high concentration contaminated grains (Metalaxyl, Parathion) artificially in lab. Diazinon was selected internal standard for GC-NPD and we confirmed pesticide residues spectrum by GC-mass. Accumulated pesticide toxicity was great dangerous, so need a rapid, accurate analysis method for grains. To get a better result, we used variation of time and wetting temperature, and recovery was not bad. According to kinds of grains, time, temperature and pesticide, different results were shown. So we have to more investigate a lot of kinds grains and pesticide for civil health and generation was used to consume great amounts grain daily

[PD4-6] [ 04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3] ]

## Chiral separation of β2-agonists by Capillary electrophoresis using β-cyclodextrins as chiral selectors in a polyethylene glycol gel.

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Chiral separation method of the enantiomers of several β2-agonists was investigated by capillary electrophoresis employing β-cyclodextrin, carboxymethyl-β-cyclodextrin, hydropropyl-β-cyclodextrin as chiral selectors. The effect of concentration of the β-cyclodextrin derivatives added to the background electrolyte, of the pH of the buffer and of the temperature on the effective mobility and resolution of the studied compounds were examined. On the basis of the result, the effects of the addition of polyethylene glycol on enantiomeric resolution could be explained. Optimum conditions of capillary electrophoretic enantioseparations with cyclodextrin additives are given.

[PD4-7] [ 04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3] ]

## Chiral separation of β-blockers after derivatization with (-)-MTPACl by Gas Chromatography/Mass Spectrometry

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Gas chromatography/mass spectrometric chiral separation method was investigated. Several β-blocking drugs were reacted with the chiral acylating reagent (-)-α-methoxy-α-(trifluoromethyl) phenylacetyl chloride to obtain, in each case, two diastereomeric amide derivatives. Prior to N-acylation, hydroxyl groups of drugs were derivatized with N-methyl-N-(trimethylsilyl) trifluoroacetamide. The effect of reaction time, reaction temperature and concentration of chiral reagent, (-)-MTPACl on the chiral derivatization reaction were investigated.
Elution order of the diastereomeric amides was observed for four compounds, atenolol, metoprolol, betaxolol and bisoprolol, by chromatographing the individual enantiomers separately under the same chromatographic condition. Each enantiomer was prepared by semi-preparative HPLC using a chiralcel OD chiral column.

[PD4-8] [ 04/21/2000 (Fri) 14:50 - 15:50 / [1st Fl, Bldg 3] ]

## Determination of optical purity in levofloxacin preparations

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Levofloxacin is the (-)-isomer of ofloxacin. Ofloxacin was originally marketed as a racemate; i.e., a mixture of the two optical isomers in equal ratios. And (-)-isomer of ofloxacin was more potent than the (+)-isomer in antibacterial activity. Therefore, levofloxacin has been developed as a single isomeric preparation by racemic switch. In this study, we separated the ofloxacin enantiomers using mobile phase additives, derivatization and a chiral stationary phase in order to control the quality of levofloxacin preparations.
Firstly was the separation carried out on a novapak C18 column using 1 mmol/L cupric sulfate-methanol (90 : 9) as a mobile phase with UV detection. Secondly was it carried out on a novapak C18 column using 0.2 mmol/L phosphoric acid (pH 1.85)-acetonitrile (80 : 20) as a mobile phase with fluorescence detection, having been derivatized with diphenylphosphinylchloride and L-leucinamide. Thirdly was it carried out on a bovineserumalbumin column using 0.2 mmol/L phosphate buffer (pH 8.0)-methanol (97 : 3) as a mobile phase with UV detection. With the result of purity determination in levofloxacin preparations, we have concluded that optical purity was quite satisfactory in levofloxacin preparations.