Letosteine is degraded in aqueous solution, its content is tested and calculated by non-aqueous titration. Analysis by HPLC is used popularly for the accuracy and precision. Especially, at low concentration, the analysis by HPLC is mor accurate than non-aqueous titration. So, we studied the analysis of Letosteine by HPLC. And the stability in test solution was carried out during 18hours.

The Letosteine was chromatographed by using C18 column, the mobile phase (sodium acetate anhydrous 2.72g, Acetic acid glacial 30ml and acetonitrile 100ml up to 1000ml with demineralized water) and UV detector (254nm) at a flow rate 0.6ml/min.

The calibration plot obtained using UV detector was linear over the range of 20 %- 120% with correlation coefficient of 0.999 (log-log scale). Reproducibility studies gave relative standard deviations of about 1 %. And Letosteine in test solution is stable within 18hours.

These results showed that analysis by HPLC is useful to assay the content of Letosteine.

[PD4-9] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Chiral purity test of (R)-(-)-salbutamol by capillary electrophoresis using sulfated-β-cyclodextrin as a chiral selector

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Chiral separation of salbutamol was investigated by capillary electrophoresis employing a sulfated-β-cyclodextrin (sulfated-β-CD). The effects of the concentration of sulfated-β-CD added to the background electrolyte and of the pH of the buffer on the effective mobility and resolution of the studied compounds were examined. Very good resolution was achieved.
Two methods for the optical purity testing of (R)-(-)-salbutamol were developed, namely capillary electrophoresis using sulfated-β-CD and high-performance liquid chromatography using chiral stationary phase. Validation data such as linearity, recovery, detection limit, and precision of the two methods are also presented. There was generally good agreement between the HPLC and CE results. These methods were found to be applicable as a practical quality control method for the enantiomeric purity determination of (R)-(-)-salbutamol.

[PD4-10] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Simultaneous enantioseparation of isoproterenol and etilefrine using derivatization by gas chromatography-mass spectrometry (GCMS)

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Simultaneous enantioseparation of isoproterenol and etilefrine using derivatization by gas chromatography-mass spectrometry (GCMS)

The art of enantioseparation has seen a most dramatic development and progress over the last two decades, maturing from a speciality field of a few experts to an area of major scientific and economic interest. In some cases of chiral drugs, One of the enantiomers has side effects and even toxic effects biologically. Mostly, Chiral HPLC has been used for isoproterenol and etilefrine to enantioseparate and determinate.
In this experiment, for simultaneous enantoseparation of isoproterenol and etilefrine, Derivatization with Trimethylchlorosilane (TMCS) and S-(-)-Trifluoroacetyl-prolyl chloride (TPC) were carried out subsequently

and injected for GCMS including achiral stationary phase. In the derivatization procedure, Both racemates were silylated and formed diastereomeric cerivatives.
As a result, It was possible to simultaneous enantioseparate both racemates by GCMS even with achiral capillary column. Compared with that of chiral HPLC, This method was found to give a better resolution and sensitivity. Furthermore, The GCMS system allows us to determinate the trace amount of it by using a SIM (single ion monitoring) mode.

[PD4-11] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Rapid analysis of tizanidine in human plasma by gas chromatography/mass spectrometry

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An efficient gas chromatography-mass spectrometry (GC-MS) method has been developed and validated for the quantitative determination of tizanidine in human plasma. Plasma samples were simply extracted with ethyl acetate at basic pH and the extracts were converted into trimethylsilyl (TMS) derivatives for the direct separation by GC-MS with the selected ion monitoring (SIM) mode. Reaction of tizanidine with N-methyl-N-(trimethylsilyl)trifluoroacetamide (MSTFA) caused di-trimethylsilylation in imidazoline moiety and this silylation significantly improved the chromatographic properties of the compound. The determination of tizanidine was accurate and reproducible, with a limit of quantitation of 0.5 ng/ml in plasma. The standard calibration curve for tizanidine was linear (r2 = 0.999) over the concentration range 0.5-10.0 ng/ml in human plasma. The intra- and inter-day precision over the concentration range of tizanidine was well within the 6.9% (relative standard deviation, RSD) and accuracy was between 99.2 and 110.5%.

[PD4-12] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Determination of some Acidic Drugs with Ion-Selective Membrane Electrode

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A sensitive poly(vinyl chloride) membrane electrode for determining the acidic drugs is described. The sensing membrane of the electrode consists of acidic drug-metal ion(II)-di-2-pyridyl ketone ternary complex as an ion-exchanger and o-nitrophenyl ether group as a plasticizer.
It shows a linear response towards mefenamate ion and ibuprofen anion over the concentration range 1× 10-2 ~ 5×10-5 mol L-1 with an anion slope of -56.3 and 54.2 ㎷ decade-1 in pH 8.9 buffer and pH 5 buffer solution respectively.
The behavior of the electrode is considerably influenced by the plasticizer employed and the optimum response appears to result when benzyl-2-nitrophenyl ether is present.
The electrode was applied to the determination of mefenamic acid and ibuprofen in pure form and in pharmaceutical preparations.

[PD4-13] [04/19/2002 (Fri) 10:00 - 13:00 / Hall E]

Postmortem Blood Concentration of Levomepromazine, Chlorpromazine, Flurazepam, Tramadol, Benztropine and Caroverine in a Case

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This paper presents a case related to levomepromazine, chlorpromazine, flurazepam, tramadol, benztropine