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Because of the differences in biological and pharmacological properties between enantiomers of chiral acidic non-steroidal antiinflammatory drugs (NSAIDs) in human body, accurate determinations of their optical purities have been in great need. Racemic ibuprofen, tiaprofen, suprofen, flubiprofen and napoxen were reacted with (1R, 2S, 5R)-(-)-menthol to convert them to corresponding diastereomeric (1R, 2S, 5R)-(-)-menthyl esters. The resulting derivatives were extracted with hexane after adjustment to pH>11, evaporated to dryness under gentle nitrogen stream and reconstituted in toluene for the direct analysis by achiral capillary column gas chromatography. Optimization of the present method and its application to chiral separation of ibuprofen and its metabolites in urine will be discussed.

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#### Determination of Histamine in a Pharmaceutical Preparation after Clean-Up by Solid-Phase Extraction

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A human immune globulin preparation (histobulin?) are made to bind a small amount of histamine (0.15 ug) to the protein (12 mg) to increase the resistance of histamine susceptibility in the treatment of allergic diseases. Strict control of histamine content of the drug are required since intake of histamine might result in hypo- or hypertension, headache, or anaphylactic shock syndromes. HPLC analytical method with pre-column fluorescent derivatization after clean-up by solid-phase extraction (SPE) was developed. Sample was applied to a polystyren-divinylbenzene (PS-DVB) SPE cartridge followed by solvent washings and eluting histamine with 100 mM NaAc in 80% methanol successively. An aliquot of the extract was pre-labeled with o-phthaldehyde-mercaptoethanol (OPA-ME) for fluorescence detection (Ex 340- Em 450 nm) in HPLC. HPLC analysis was performed under the conditions: Eluent with 35% AcCN in 50 mM phosphate(pH 6.8) showed the retention time of the analyte and internal standard (3-methylhistamine) at 7.0- and 10.8 min., each respectively on a phenyl column. Histamine was recovered quantitatively afforded 80% in 0.1 ppm range with efficient removal of proteins and glycine by the SPE method. Good linearity was obtained up to 10 ug/mL of histamine. Quantitation range was between 0.01 and 0.5 ug/mL for the sample. The method was successfully applied to determine micro-amount of histamine in the pharmaceutical

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#### Studies on the Analysis of Anti-impotent Drugs

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Sildenafil citrate(SC, Viagra<sup>®</sup>) is the oral treatment for erectile dysfunction. Vardenafil hydrochloride, tadalafil and modified sildenafil have anti-impotent effect and have possibility to be used illegally. The aim of this study was to set up simultaneous determination method of anti-impotent drugs using TLC and HPLC. The anti-impotent drugs were extracted with 50% methanol and then diluted with mobile phase for HPLC and also extracted with developing solvents as sample solutions for TLC. The sample solutions for TLC was applied to Silica gel F<sub>254</sub> plates with chloroform/ethanol(9:1) as developing solvents. Spots were located under UV radiation at 254nm and visualized with iodine vapor. The HPLC analysis was carried out reverse phase of CAPCELL PAK C<sub>18</sub>(4.6×250mm, 5μm) with 0.1% SHS in 0.1% phosphoric acid/acetone(nitrile)(73:27) as mobile phase and the eluate was detected by a photo-diode array. This method can be used in detecting the fake Viagra<sup>®</sup> and other anti-impotent drugs illegally used in the products.