[*Heterocycles.*, **51**, 2415-2421 (1999)] [Lab. of Manufacturing Pharmacy]

Convenient Synthesis of Cyclohexa[*a*]pyrrolo[2,1-*b*][3]benzazepine, A Cephalotaxus Alkaloid Analogue.

Hitoshi TANAKA, Mitsunobu DOI, Hiroshi SHIMIZU, * and Hideo ETOH

Irradiation of *N*-(3-hydroxy-4-methoxyphenethyl)-3-(2-iodo-5-methoxyphenyl)propionamide in methanol in the presence of sodium hydroxide furnished 3-hydroxy-2,12-dimethoxy-6,7,9,10-tetrahydrodibenz[*d,f*]azecin-8(5*H*)-one which was successfully led to cephalotaxine analogue bearing pyrrolo[2,1-*b*][3]benzazepine skeleton by reduction with borane followed by Birch reduction and subsequent acidic treatment. The structure of the cephalotaxine analogue was established by means of X-Ray crystallography.

[*Phytochem. Anal.*, **10**, 247-253 (1999)] [Lab. of Manufacturing Pharmacy]

Quantitative Analysis of All Types of β-Carboline Alkaloids in Medicinal Plants and Dried Edible Plants by High Performance Liquid Chromatography with Selective Fluorometric Detection.

Hironori TSUCHIYA, Hideaki HAYASHI, Masaru SATO, Munekazu IINUMA, and Hiroshi SHIMIZU*

A HPLC method was developed to determine all types of β-carboline alkaloids in medicinal and edible plants. The alkaloids, including internal standards, were purified from plant homogenates by serial extractions after reacting with fluorescamine. The extracts were analysed twice using reversed-phase chromatography with fluorometric detection optimized for each individual analyte. The simultaneous separation of harmol, norharman, harman, harmine, harmalol, harmaline, tetrahydronorharman, tetrahydroharman and two internal standards was achieved within 17 min by an isocratic elution. β-Carbolines, dihydro-β-carbolines and tetrahydro-β-carbolines could be quantitatively determined in concentrations of 0.01-50.0 ng/mL. Application of the proposed method has revealed that medicinal plants and dried edible plants contain β-carboline alkaloids at ng/g to μg/g levels. Norharman and harman were distributed in all the tested plants, while harmol and harmalol were found only in some plant species.

[*Chem. Pharm. Bull.*, **47**, 440-443 (1999)] [Lab. of Manufacturing Pharmacy]

Beta-Carboline Alkaloids in Crude Drugs.

Hironori TSUTHIYA, Hiroshi SHIMIZU, * and Munekazu IINUMA

Beta-carboline alkaloids in crude drugs were quantified by a reversed-phase HPLC method without interference from their artifactual formation during analysis and with fluorometric detection specific to each individual analyte. 1-Methyl-β-carboline, β-carboline, 7-hydroxy-1-methyl-β-carboline, 7-methoxy-1-methyl-β-calboline, 1-methyl-1,2,3,4-tetrahydro-β-carboline and 1,2,3,4-tetrahydro-β-carboline showed a wide distribution in the crude drugs and the former two β-carbolines were detected in all those tested. Schisandrae Fructus, Pinelliae Tuber, Evodiae Fructus and *Passiflora incarnata* contained relatively large amounts of β-carbolines at ng–μg/g dry weight levels. Beta-carboline alkaloids may be responsible for the pharmacological effects of crude drugs as the potent active substances.

[*Anal. Sci.*, **15**, 611-612 (1999)] [Lab. of Instrumental Center]

Crystal Structure of Phenolphthalein.

Hiromasa SUGIURA, Toshiyuki KATO, Hitoshi SENDA, Ko-Ki KUNIMOTO, Akio KUWAE and Kazuhiko HANAI*

Crystals of phenolphthalein are orthorhombic: space group $Pna2_1$ with a=19.276(3), b=14.822(2), c=11.3884(9)Å, and Z=8. There are two independent molecules in the asymmetric unit. Each of the three moieties (isobenzofuran ring and two *para*-hydroxyphenyl rings) is almost planar. The geometry differences between the two molecules are found in the orientations of the planes.