Separation of Lutidine Isomers by Selective Enclathration

Jacky S. Bouanga Boudiombo¹, Hong Su¹, Neil Ravenscroft¹, Susan A. Bourne¹, Edwin Weber², and Luigi R. Nassimbenia¹

¹Centre for Supramolecular Chemistry Research, Department of Chemistry, University of Cape Town, Rondebosch 7701, SouthAfrica, ²Institut für Organische Chemie, TU Bergakademie Freiberg, Leipziger Straße 29, D-09596 Freiberg/Sachs, Germany

* luigi.nassimbeni@uct.ac.za

Molecular selectivity by host-guest procedures is an increasing method to help in the separation of isomers¹. The separation of a component from a mixture may be carried out by exploiting the physico-chemical properties of the compounds in that mixture. The most common techniques, viz. distillation, crystallization, liquid–liquid extraction, and various forms of chromatography, rely on differences in solubility and vapor pressure of the components. In the case of molecular isomers, however, their macro-properties are often similar, rendering the traditional separation techniques inefficient. In such cases the process of enclathration by a suitable host compound is a useful technique.^{2,3,4}

In this study, the host compound 3,3'-bis(9-hydroxy-9-fluorenyl)-2-2'-binaphthyl, H1, has been employed to separate the six isomers of lutidine. Competition experiments showed that the preference for enclathration is in the sequence 3,4-LUT > 2,6-LUT > 2,3-LUT > 2,5-LUT > 2,4-LUT \approx 3,5-LUT. The structures yielded results that agree with the 1H NMR analyses and with the thermal analysis. The effects of mixed hosts and vapor-phase competitions were briefly explored with two extra hosts, namely, 2,2'-bis(1-hydroxy-4,5-dihydro-2:3,6:7-dibenzocycloheptadien-1-yl)biphenyl(H2) or 3,3'-bis(di-p-olylhydroxymethyl) -1,1'-binaphthyl (H3). Following this study, 2,2'bis(1-hydroxy-4,5-dihydro-2,3:6,7-dibenzocycloheptatrien-1-yl)-biphenyl, H2, was then employed to discriminate between all the pairs of lutidine isomers. The preference for guest enclathration follows the sequence 3,4-LUT>2,4-LUT \approx 3,5-LUT>2,5-LUT>2,3-LUT>2,6-LUT. This has been confirmed by guest-release endotherms measured by DSC. Four extra diol host compounds with similar structures were tested on pairs of lutidine isomers which were poorly separated by H2.

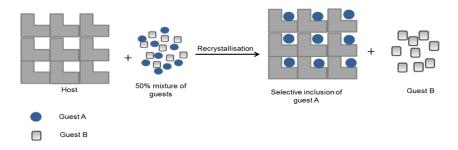


Figure 1. Molecular chemistry by host-guest chemistry.

Keywords: Dio-hosts, separation, lutidine isomers

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