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# Separation of Lutidines by Enclathration

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Three related diol host compounds have been employed to separate lutidine isomers. Thirteen equimolar mixtures of the isomers were enclathrated by each of the host compounds in turn, and the results monitored by NMR. When the selectivity was poor, crystal structures of the host compounds with each of the single isomers and their mixture, were elucidated. By appropriate use of the three host molecules, it proved possible to separate each isomer. The selectivity trend is 2,3-LUT ≈ 3,4-LUT > 2,5-LUT > 2,4-LUT > 2,6-LUT.

## Introduction

Molecular recognition is the process that gives rise to selectivity, an important aspect of supramolecular chemistry. Lehn[1] discusses this in terms of both the energy and information involved in the combining of two or more molecules to form a distinct complex. In terms of host-guest chemistry, we may formulate the reversible reaction as

$$H + nG \leftrightharpoons H \cdot Gn$$

where H is the host molecule and G is the guest which forms the host-guest complex with a guest/host ratio n.

The features which maximize the complementarity between host and guest, and give rise to the stability of the inclusion compound H·Gn, include the size and shape of the host molecule, its allowed conformations, its binding sites and their reactivity. All these properties contribute to the information content of the host and although no quantitative measure is employed for its evaluation, Lehn has developed an index to describe ligand topology, which is useful in its evaluation for the selectivity of metal ions.[2] Selectivity can be thermodynamic or kinetic in nature, and may occur in the gas, liquid or solid phase. The most useful method of testing selectivity is to expose a solid host to a mixture of liquid guests A and B:

$$H(s, \alpha) + n_1A(\ell) + n_2B(\ell)) \leftrightharpoons H{\cdot}Am_1{\cdot}Bm_2\ (s, \beta)$$

The solid host H, in its non-porous α-phase, also known as the apohost, is dissolved in a known mixture of guests and, upon recrystallization forms the inclusion compound, the enclathrating solid β-phase, whose composition is determined by suitable analytical techniques such as NMR, thermal gravimetry, or gas chromatography. The selectivity coefficient[3] of the host, is defined as

$$K_{A:B} = Z_A/Z_B \times X_A/X_B$$

where $X_A$, $X_B$ are the mole fractions of the guests A and B in the liquid mixture and $Z_A$ and $Z_B$ are their mole fractions in the crystals. When the selectivity coefficient is ≥10, the separation is deemed to be efficient. However, competition experiments yielding low values of $K_{A:B}$ can produce interesting results regarding the structures of the corresponding inclusion compounds and may give insights into the mechanism leading to the discrimination of a given guest over another.

Separation by host-guest chemistry is deemed appropriate when the liquid mixture has components with similar boiling points, which would make distillation inefficient, and we have applied this technique to the separation of lutidines. There are six isomers of lutidine, hereafter labelled LUT, and their boiling points are reported in Table 1. Lutidines as guest molecules in inclusion compounds have been studied and their structures have been elucidated. The co-crystals and salts of succinic and fumaric acids with isomers of lutidine have been characterized.[4] A systematic study of lutidinium pamoate salts, which discusses the role of the solvent on the ensuing structures, shows that the structures depend on the state of ionization of pamoate anion.[5] Diol host compounds have been employed to enclathrate lutidines and 1,1,2,2-tetraphenylethane-1,2-diol forms inclusion compounds with 2,6-, 3,5- and 3,4-lutidines. These compounds were characterized structurally and by thermal methods.[6] Competition experiments in which 2,4-LUT, 2,6-LUT and 3,5-LUT were

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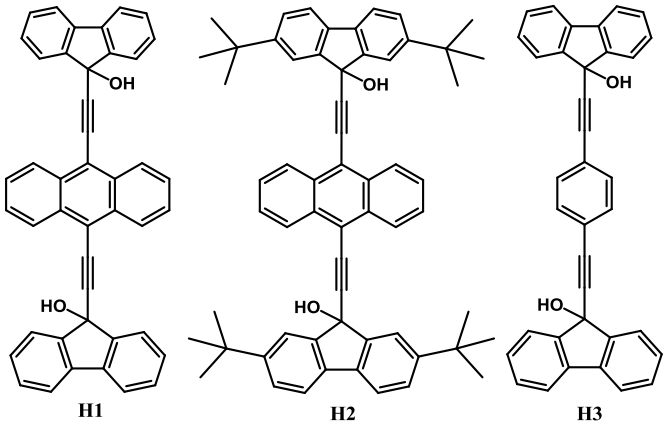
simultaneously exposed to 1,1-bis(4-hydroxyphenyl)cyclohexane, showed that the preferential selectivity was 3,5-LUT > 2,4-LUT > 2,6-LUT.[7] Similar results were obtained in these three same isomers by employing the host 1,4-bis(9-hydroxy-9-fluorenyl)benzene.[8] The host 2,2'-dihydroxy-1,1'-binaphthyl forms inclusion compounds with 2,4-LUT, 2,6-LUT, and 3,5-LUT and their kinetics of desolvation were studied by carrying out isothermal thermal gravimetry (TG) at selected temperatures. The mechanism of decomposition and accompanying energies of activation were evaluated.[9]

We have chosen the host compounds 9,10-bis[2-(9-hydroxy-9-fluorenyl)ethynyl]anthracene, H1, 9,10-bis[2-(2,7-di-tert-butyl-9-hydroxy-9-fluorenyl)ethynyl]anthracene, H2 and 1,4-bis[2-(9-hydroxy-9-fluorenyl)ethynyl]benzene, H3 as shown in Scheme 1. In this procedure a mixture of lutidines was exposed to a selected host compound that forms a crystalline host-guest complex, which was separated from the mother liquor. The entrapped lutidine guest could then be released from the crystals by gentle warming and the host compound recycled. For this process to be successful, the host should be selective to one particular guest in the mixture, but the discrimination is seldom completely efficient, requiring the procedure to be repeated several times in order to obtain satisfactory purity of the targeted isomers.




**Scheme 1** Graphical structures of the three host compounds H1, H2 and H3

**Table 1** Boling points of isomers of lutidines

| | 2,3 LUT | 2,4 LUT | 2,5 LUT | 2,6 LUT | 3,4 LUT | 3,5 LUT |
|---|---|---|---|---|---|---|
| Bp/°C | 162 | 159 | 157 | 144 | 163 | 169 |

## Experimental

### General remarks

The melting point was determined with a Reichert hot-stage apparatus. The $^{1}H$ and $^{13}C$ NMR spectra were measured for solutions ($Me_4Si$ as internal standard, ppm) with a Bruker MSL 300 spectrometer. The mass spectrum was obtained using an A.E.I. MS-50 instrument. The microanalysis was carried out on a Heraeus CHN rapid analyzer. Solvents were dried by standard procedure.

### Synthesis

The starting compound 9,10-dibromoanthracene as well as the components of the catalyst were purchased from commercial sources. 2,7-di-*tert*-butyl-9-ethynylfluoren-9-ol was prepared as described.[10] The host compounds H1[10] and H3[11] were synthesized as reported in the literature.

*Preparation of H2*

To a stirred and boiling solution of 9,10-dibromoanthracene (0.52 g, 2.0 mmol) in trimethylamine-toluene (75 ml, 2:1, v/v, dried and degassed), 2,7-di-*tert*-butyl-9-ethynylfluoren-9-ol (1.59 g, 5.0 mmol) was added under argon. After cooling the solution to room temperature, Pd(II) acetate (25 mg), triphenylphosphane (75 mg) and Cu(I) iodide (25 mg) were added. The mixture was heated to 90 °C until the reaction was complete (about 4 h, tested by thin-layer chromatography) and was then cooled to room temperature. The catalyst and the triethylammonium salts were filtered off and washed with diethyl ether (25 ml). The filtrate and washings were evaporated under reduced pressure. The residue was dissolved in diethyl ether and washed (diluted hydrochloric acid, aqueous $NaHCO_3$ and $H_2O$, in this sequence). The organic layer was separated, dried ($Na_2SO_4$) and evaporated. The remaining solid was treated with MeOH, then collected and crystallized from toluene to yield colorless crystals (1.2 g, 74%). Mp 248 °C (from toluene). NMR data: $\delta_H$ (300 MHz, $CDCl_3$) 1.43 (36H, s, $CH_3$), 2.27 (2H, s, OH), 7.27-7.54 (12H, m, Ar-H), 8.18 (4H, d, Ar-H), 8.70 (4H, d, Ar-H). $\delta_C$ (75 MHz, $CDCl_3$) 31.53 ($CH_3$), 35.16 (C-$CH_3$), 77.31 (C-OH), 79.32 (C≡C), 100.50 (C≡C), 119.77, 121.36, 125.08, 126.77, 126.80, 128.56, 132,51, 134.08, 136.56, 147.27 Ar-C). MS data (EI) calc. for $C_{60}H_{58}O_2$ (811.12), found: 811.1 *m/z* $[M]^+$. Anal. calc. for $C_{60}H_{58}O_2$: C, 90.46; H, 4.29, found: C, 90.31; H, 4.21%.

### X-ray diffraction

Crystal data for inclusion compounds H1·2(2,3-LUT), H1·2(3,4-LUT), H2·2(2,3-LUT), H2·2(3,4-LUT), H3·2(3,4-LUT) and H3·2(2,3-LUT) were collected on a Nonius Kappa CCD diffractometer with graphite monochromated Mo-$K_\alpha$ radiation (λ = 0.71073 Å) at 173 K using an Oxford Cryostream 600. The strategy for the data collection was evaluated using COLLECT software,[12] and intensity data were scaled and reduced using the program DENZO-SMN.[13] The Crystal data for inclusion compounds H1·2(2,3-LUT/3,4-LUT) and H2·2(2,3-LUT/3,4-LUT) were collected on a Bruker DUO APEX II diffractometer with graphite-monochromated Mo-$K_\alpha$ radiation (λ = 0.71073 Å) at 173 K using an Oxford Cryostream 700. Data reduction and cell refinement were performed using SAINT-Plus.[14] The space groups were determined from systematic absences by XPREP[15] and further justified by the refinement results. The structures were solved by direct methods and refined by full-matrix least-squares on $F^2$ using SHELX-97 program package[16] with the aid

of the interface program X-Seed.[17] The hydrogen atoms bound to carbon atoms were placed at idealized positions and refined as riding atoms with $U_{iso}$ (H) = 1.2 $U_{eq}$ (Ar-H) or 1.5 $U_{eq}$ ($CH_3$). The hydroxyl hydrogens were located in difference electron density maps and refined independently. CCDC deposit numbers 1418275-1418282 contain the supplementary crystallographic data for this paper.

## Results and Discussion

The initial survey was carried out with binary equimolar mixtures of the lutidines and the single host compounds H1, H2 and H3 in turn. We analyzed the results by NMR spectroscopy. These are shown in Tables 2, 3 and 4. When the mole fraction of a given isomer in the host-guest complex was >85%, we regarded this as 'complete' selectivity and did not investigate the compound further. The results are indicated by the lutidine substituent numbers in the squares.

**Table 2** Selectivity exhibited by H1 as confirmed by $^1$H NMR

| Lutidine | 2,3 | 2,4 | 2,5 | 2,6 | 3,4 | 3,5 |
|---|---|---|---|---|---|---|
| 2,3 | | 2,3 | 2,3 | 2,3 | 2,3=0.62<br>3,4=0.38 | 2,3 |
| 2,4 | | | 2,5 | | 3,4 | 3,5 |
| 2,5 | | | | 2,5 | | 3,5 |
| 2,6 | | | | | 3,4 | 3,5 |
| 3,4 | | | | | | 3,5 |
| 3,5 | | | | | | |

For six lutidine isomers, there are fifteen different equimolar mixtures. Where the NMR signals of the lutidines overlapped with those of the host compounds or with each other, we omitted the experiments, and this is shown as hashed squares in Tables 2, 3 and 4 (See ESI Table S1). From Table 2, the result of the poorest selectivity is that between 2,3-LUT and 3,4-LUT, which resulted in the enclathration of 62% (2,3-LUT) and 38% (3,4-LUT) as measured by NMR. The crystal structures of H1·2(2,3-LUT), H1·2(3,4-LUT) and H1·2,3-LUT/3,4-LUT were elucidated.

The crystal data and refinement parameters for the structures are given in Table 5. The structure H1·2(2,3-LUT) crystallizes in the space group $P2_1/n$ with Z = 2. The host is located at a centre of inversion at Wyckoff position a. The 2,3-LUT guests are hydrogen bonded to the host via (Host)-OH···N(2,3-LUT) bonds with O···N = 2.811(1) Å. The packing is shown in Figure 1. The metrics of this and all the other hydrogen bonds occurring in subsequent structures are given in the ESI (Table S3).

The structure of H1·2(3,4-LUT) crystallizes in *P*-1 with Z = 1. The host is again located in a centre of inversion at Wyckoff position c. This structure is also stabilized by (Host)-OH···N(3,4-LUT) hydrogen bonds with d(O···N) = 2.791 (2) Å.

The structure of H1·2(2,3-LUT/3,4-LUT) is isomorphous with that of H1·2(2,3-LUT), in that the host atoms are located in the same positions in both structures. However, the guest is composed of both the 2,3-LUT and the 3,4-LUT at the same site, but with site occupancy factors that refined to 0.63 for the 2,3-LUT and 0.37 for the 3,4-LUT. These are close to the values obtained in the NMR experiment. The geometry of the two disordered guests is shown in Figure 2.

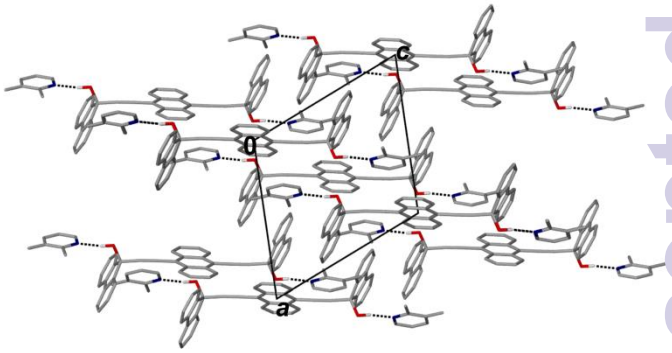



**Figure 1** The packing arrangement of H1·2(2,3-LUT) viewed down *b*-axis

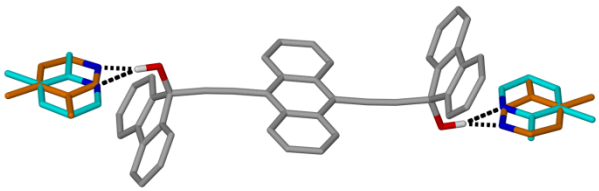


**Figure 2** The asymmetric unit of H1·2(2,3-LUT/3,4-LUT) showing the presence of 2,3-LUT and 3,4-LUT. Color code; 2,3-LUT (green) and 3,4-LUT (brown)

We repeated the NMR analysis with second host molecule H2, which yielded the results shown in Table 3. Again the only mixture which displayed poor selectivity was that between 2,3-LUT and 3,4-LUT. We therefore pursued the structural investigation of the three relevant structures.

The structure of H2·2(2,3-LUT) crystallizes in the space group *P*-1 with Z = 1. The host molecule lies on a centre of inversion at Wyckoff position e and the 2,3-LUT guest is disordered with site occupancies of 0.5. Both display hydrogen bonds with d(O···N) = 2.796(3) Å and 2.769(5) Å, respectively. The *tert*-butyl groups in the host are not disordered. The structure of H2·2(3,4-LUT) also crystallizes in *P*-1 with Z = 1. This structure displays the hydroxyl moieties of the host molecule in the *cis*-conformation and pairs of these hosts stack across a centre of inversion at Wyckoff position b. The

hydroxyl moieties form hydrogen bonds with one of the guest lutidines, as well as to the hydroxyl oxygen of a neighbouring host, forming a $R^2_2(26).D_1^1(3)$ hydrogen bonded system.[18] This is shown in Figure 3. The second 3,4-LUT guest is disordered and is not hydrogen bonded to the host.

**Table 3** Selectivity exhibited by H2 as confirmrd by $^1$H NMR

| Lutidine | 2,3 | 2,4 | 2,5 | 2,6 | 3,4 | 3,5 |
|---|---|---|---|---|---|---|
| 2,3 | | 2,3 | 2,3 | 2,3 | 2,3=0.59<br>3,4=0.41 | 2,3 |
| 2,4 | | | 2,5 | | 3,4 | 3,5 |
| 2,5 | | | | 2,5 | | 2,5=0.80<br>3,5=0.20 |
| 2,6 | | | | | 3,4 | 3,5 |
| 3,4 | | | | | | 3,4 |
| 3,5 | | | | | | |

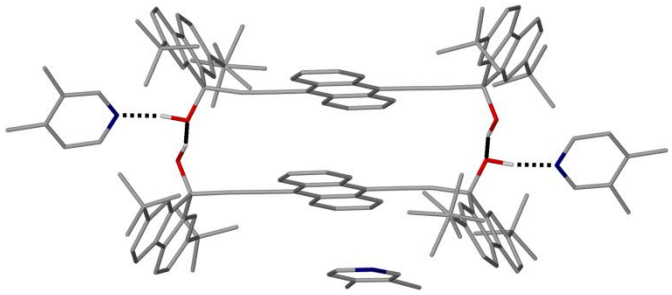


**Figure 3** $R^2_2(26).D_1^1(3)$ hydrogen bonding pattern observed in H2·2(3,4-LUT)

The structure of the H2·2(2,3-LUT/3,4-LUT) has cell dimensions similar to those of the H2·2(2,3-LUT) structure, but the two guests were disordered and their modelling eventually yielded two distinct moieties. One was of pure 2,3-LUT and the other was a mixture of 2,3-LUT and 3,4-LUT as shown in Figure 4. The overall site occupancy factors were 0.69 for 2,3-LUT and 0.31 for 3,4-LUT, in fair agreement with the NMR determination.

Following the philosophy of the Dutch resolution method,[19] we combined H1 and H2 in equimolar proportions and dissolved these in an equimolar mixture of 2,3-LUT and 3,4-LUT. This experiment yielded two types of crystals which could be distinguished by inspection with an optical microscope. One group of crystals were block like with dimensions varying from 0.2 to 0.3 mm. These were carefully separated from the mixture and their NMR spectrum recorded, yield 64% (2,3-LUT) and 36% (3,4-LUT). A suitable specimen was submitted for X-ray diffraction and its structure proved to be that of H1·2(2,3-LUT/3,4-LUT) with site occupancy factors of 60% and 40% for 2,3-LUT and 3,4-LUT respectively.

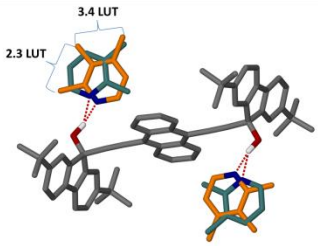



**Figure 4** A perspective view of asymmetric unit of H2·2(2,3-LUT/3,4-LUT)

This result is practically identical to the elucidated structure. The second group of crystals in the mixture were very small and unsuitable for X-ray analysis. Their separation from the mixture was difficult and time consuming. Their NMR spectrum showed them to be H2·2(2,3-LUT/3,4-LUT) with 61% of 2,3-LUT and 39% of 3,4-LUT, a result similar to that previously obtained. The separation experiment is summarized in Figure 5 (see Table S2 and Figure S2 ESI).

The NMR analysis of the third host H3 is shown in Table 4. Interestingly this host completely selects 3,4-LUT over 2,3-LUT. We therefore investigated the two structures of H3 with these lutidines. H3·2(3,4-LUT) crystallizes in *P*-1 with Z = 1. The host molecule is located on a centre of inversion at Wyckoff position c and the 3,4-LUT is bonded to the host via a (Host)-OH···N(Guest) hydrogen bond.

**Table 4** Selectivity exhibited by H3 as confirmed by $^1$H NMR

| Lutidine | 2,3 | 2,4 | 2,5 | 2,6 | 3,4 | 3,5 |
|---|---|---|---|---|---|---|
| 2,3 | | 2,3 | 2,3 | 2,3 | 3,4 | 3,5 |
| 2,4 | | | 2,4=0.76<br>2,5=0.24 | | 3,4 | 3,5 |
| 2,5 | | | | 2,5 | | 3,5 |
| 2,6 | | | | | 3,4 | 3,5 |
| 3,4 | | | | | | 3,5 |
| 3,5 | | | | | | |

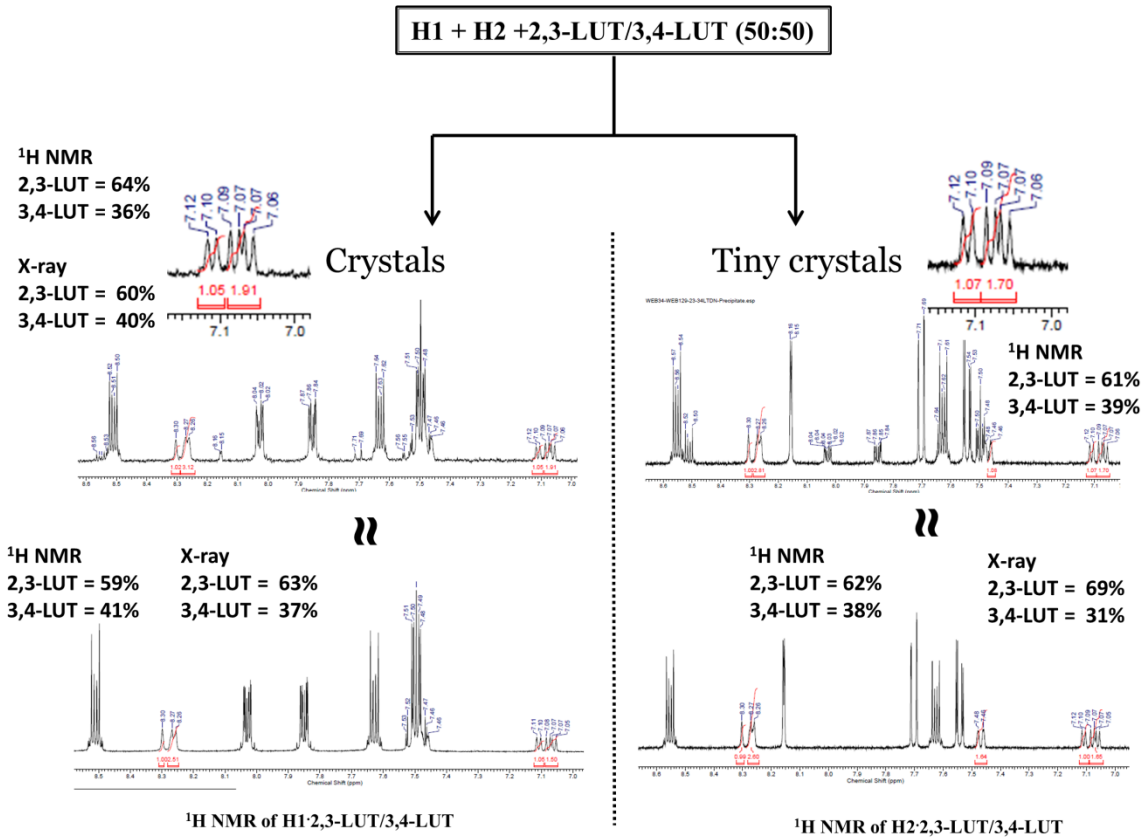



**Figure 5** [1]H NMR result of separation expertiment carried out by the combination H1 and H2 with equimolar mixture of 2,3-LUT/3,4-LUT

The H3·2(2,3-LUT) compound also crystallizes in *P*-1 with Z = 1, with H3 located at Wyckoff position h. The guest is ordered and hydrogen bonded to the host and the metrics of the two hydrogen bonds are given in Table 6. The packing is shown in Figure 6. The question arises as to why H3 selects 3,4-LUT over 2,3-LUT. We therefore carried out a packing analysis using the program CrystalExplorer,[20] and fingerprint plots were generated for both structures, using the guest molecules as targets. The results of the 2D plots are

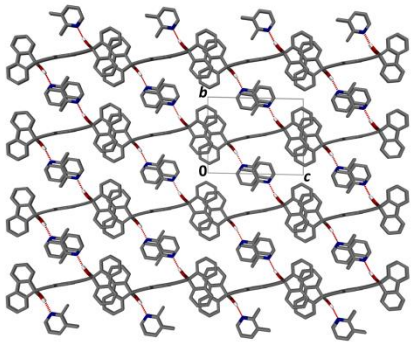



**Figure 6** The packing arrangement of crystal structure of H3·2(2,3-LUT) viewed down *a*-axis

similar and the percentages of the non-bonded interactions were not significantly different. These plots and the accompanying numerical data have been deposited as supplementary information (Figure S1). We therefore considered the difference in the hydrogen bonding in the two structures, the metrics of which are shown in Table 6. We employed the potential of Vedani and Dunitz[21] which has the form

$$V(\text{H-bond}) = (A/R^{12} - C/R^{10}) \cos^2\theta$$

where R is the distance between the donor hydrogen and the acceptor atom and θ is the donor-H···acceptor angle. The $\cos^2\theta$ term is the energy penalty paid by the bond in order to take non-linearity into account. The calculation yielded an H-bond energy of 14.69 $kJmol^{-1}$ for H3·2(2,3-LUT) and 15.06 $kJmol^{-1}$ for H3·2(3.4-LUT). The stronger H bond for the latter structure is also in agreement with the CrystalExplorer results for the (Host)-OH···N(Guest) percentage interaction which is 7.7% for 3,4-LUT and 7.2% for 2,3-LUT. Although the

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**Table 5** Crystallographic data for the inclusion compounds

| Compound | H1.2(2,3-LUT) | H1.2(3,4-LUT) | H1.2(2,3-LUT/3,4-LUT) | H2.2(2,3-LUT) | H2.2(3,4-LUT) | H2.2(2,3-LUT/3,4-LUT) | H3.2(2,3-LUT) | H3.2(3,4-LUT) |
|---|---|---|---|---|---|---|---|---|
| **Structure** | **I** | **II** | **III** | **IV** | **V** | **VI** | **VII** | **VIII** |
| Comment | | | Similar to I | | | Similar to IV | | Obtained from H3 and 2,3-LUT/3,4-LUT |
| Empirical formula | $C_{58}H_{44}N_2O_2$ | $C_{58}H_{44}N_2O_2$ | $C_{58}H_{44}N_2O_2$ | $C_{74}H_{76}N_2O_2$ | $C_{141}H_{125}N_3O_4$ | $C_{74}H_{74.72}N_2O_2$ | $C_{50}H_{40}N_2O_2$ | $C_{50}H_{40}N_2O_2$ |
| Formula weight | 800.95 | 800.95 | 800.95 | 1025.37 | 1925.44 | 1024.08 | 700.84 | 700.84 |
| Crystal system | monoclinic | triclinic | monoclinic | triclinic | triclinic | triclinic | triclinic | triclinic |
| Space group | $P2_1/n$ | $P$-1 | $P2_1/n$ | $P$-1 | $P$-1 | $P$-1 | $P$-1 | $P$-1 |
| $a$ [Å] | 11.8675(4) | 9.780(2) | 11.8056(14) | 8.870(2) | 10.964(2) | 8.8150(18) | 7.7450(4) | 7.6847(3) |
| $b$ [Å] | 15.6072(4) | 11.388(2) | 15.6491(19) | 10.410(3) | 14.240(3) | 10.541(2) | 10.1392(4) | 10.8142(5) |
| $c$ [Å] | 12.2828(4) | 11.566(2) | 12.2647(15) | 16.836(5) | 20.271(4) | 16.794(3) | 12.8039(6) | 12.3664(5) |
| $\alpha$ [°] | 90 | 115.12(3) | 90 | 76.160(5) | 109.78(3) | 75.30(3) | 87.030(2) | 77.867(2) |
| $\beta$ [°] | 112.860(1) | 101.58(3) | 111.724(2) | 88.162(6) | 97.99(3) | 88.23(3) | 74.328(2) | 77.131(2) |
| $\gamma$ [°] | 90 | 104.11(3) | 90 | 76.165(6) | 95.00(3)) | 76.92(3) | 74.741(2) | 70.639(2) |
| $V$[Å$^3$] | 2096.32(11) | 1061.1(6) | 2104.9(4) | 1465.1(7) | 2918.3(10) | 1469.7(6) | 933.76(7) | 934.78(7) |
| $Z$ | 2 | 1 | 2 | 1 | 1 | 1 | 1 | 1 |
| Temperature(K) | 173 | 173 | 173 | 173 | 173 | 173 | 173 | 173 |
| $D_{calc}$(g/cm3) | 1.269 | 1.253 | 1.264 | 1.162 | 1.095 | 1.157 | 1.246 | 1.245 |
| $\mu$(Mo-Kα)(mm$^{-1}$) | 0.076 | 0.075 | 0.076 | 0.068 | 0.065 | 0.068 | 0.075 | 0.075 |
| $F$(000) | 844 | 422 | 844 | 550 | 1024 | 549 | 370 | 370 |
| Crystal size | 0.21x0.17x0.12 | 0.29x0.28x0.06 | 0.33x0.18x0.16 | 0.23x0.06x0.03 | 0.31x0.21x0.10 | 0.39x0.11x0.04 | 0.23x0.19x0.11 | 0.32x0.14x0.11 |
| Reflections collected | 9392 | 9641 | 17168 | 11294 | 24990 | 13994 | 8018 | 7992 |
| Independent reflections | 4794 | 4837 | 5396 | 5589 | 13238 | 5626 | 4249 | 4239 |
| Observed reflections [$I$>2s($I$)] | 3201 | 3361 | 2989 | 2788 | 8294 | 2901 | 3101 | 3354 |
| Parameters | 283 | 282 | 277 | 429 | 668 | 351 | 247 | 247 |
| Goodness-of-fit | 1.021 | 1.040 | 1.031 | 0.966 | 1.029 | 1.028 | 1.037 | 1.023 |
| $R_1$[$I$>2s($I$)][a] | 0.0448 | 0.0503 | 0.0706 | 0.0606 | 0.0702 | 0.0739 | 0.0425 | 0.0407 |
| $wR_2$(all data)[b] | 0.1030 | 0.1284 | 0.1391 | 0.1323 | 0.1942 | 0.1854 | 0.1027 | 0.0974 |

**Table 6** Metrics of the hydrogen bonds for H3·2(2,3-LUT) and H3·2(3,4-LUT)

| | H3·2(2,3-LUT) | H3·2(3,4-LUT) |
|---|---|---|
| d(O-H)/Å | 0.971 | 0.971 |
| d(H···N)/Å | 1.851 | 1.823 |
| d(O···N)/Å | 2.789 | 2.792 |
| θ = (O-H··· N)/° | 161.61 | 176.9 |

differences are small, they are consistent with the hydrogen bonding favoring the selection of 3,4-LUT by host H3, probably due to steric hindrance of the methyl group at the ortho-position of the 2,3-LUT.

## Conclusions

Three structurally similar diol host compounds have been employed for the separation of lutidine isomers. 13 of 15 equimolar mixtures of pairs of the six lutidine isomers were crystallized with each of the three host molecules. In two cases where selectivity for a given lutidine isomer was poor, the crystal structures of the host compound with each isomer and the mixture of isomers were elucidated. By appropriate use of the three host molecules it is possible to separate the isomers of lutidine and the selectivity trend is 2,3-LUT ≈ 3,4-LUT > 2,5-LUT > 2,4-LUT > 2,6-LUT.

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## Separation of Lutidines by Enclathration

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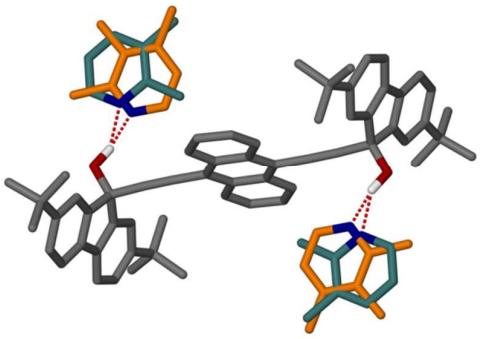

The selectivity of lutidine isomers by diol host compounds follows the trend: 2,3-LUT ≈ 3,4-LUT > 2,5-LUT > 2,4-LUT > 2,6-LUT.