A topochemical rearrangement with multiple inversions of configuration

Saul Wolfe, Yih-Huang Hsieh, Raymond J. Batchelor, Frederick W.B. Einstein, and Ian D. Gay

Abstract: Crystalline 2-benzyloxypyridine-1-oxide rearranges slowly at room temperature to crystalline 1-benzyloxy-2pyridone. No intermediates are detected when the process is followed by solid-state ¹³C NMR. The crystal structure of the pyridine-1-oxide strongly suggests that a topochemically controlled intramolecular process, in which the benzyl group migrates with retention of configuration, is not feasible. On the other hand, although somewhat disfavoured by initial solid-state O···C···O angles significantly less than the ideal 180°, intermolecular topochemically controlled processes can be envisaged that lead, with multiple inversions of configuration, either to net retention of configuration or to net inversion of configuration in the benzyl group. In contrast to the 50–80% inversion observed in solution, in the solid state only inversion is observed experimentally when chirally labelled α -deuteriobenzyloxypyridine-1-oxide is allowed to rearrange.

Key words: X-ray crystallography, solid-state ¹³C NMR, benzyl- α -D-alcohol, 2-benzyloxypyridine-1-oxide, 1-benzyloxy-2-pyridone.

Résumé : Le 1-oxyde de 2-benzyloxypyridine à l'état cristallin se réarrangement lentement, à la température ambiante, pour conduire à la 1-benzyloxypyrid-2-one. Lorsqu'on a suivi cette transformation par RMN du ¹³C à l'état solide, on n'a pas pu détecter d'intermédiaires. La structure cristalline du 1-oxyde de pyridine suggère fortement qu'il n'est pas possible de suggérer l'existence d'un processus intramoléculaire contrôlé de façon topochimique dans lequel le groupe benzyle migrerait avec rétention de configuration. Par ailleurs, même s'il est légèrement défavorisé à l'état solide par des angles initiaux O···C···O inférieurs à la valeur idéale de 180°, il est possible d'envisager des processus intermoléculaires contrôlés de façon topichimique qui conduiraient après de multiples inversions de configuration à une rétention ou à une inversion nette de configuration dans le groupe benzyle. Par opposition aux degrés d'inversion de 50 à 80% observés expérimentalement en solution, le réarrangement du 1-oxyde de l' α -deutérobenzyloxypyridine à l'état solide ne donne lieu qu'à de l'inversion.

Mots clés : diffraction des rayons X, RMN du ¹³C à l'état solide, alcool α -D-benzylique, 1-oxyde de 2-benzyloxypyridine, 1-benzyloxypyrid-2-one.

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We recently reported (1) that the Tieckelmann reaction, the conversion of a 2-alkoxypyridine-1-oxide (1) into a 1-alkoxy-2-pyridone (2) (2), is not an intramolecular [1s,4s] sigmatropic rearrangement (3), as some workers had previously believed (4), but a two-step intermolecular process. In the first step (see Fig. 1), an alkyl group R* is transferred, with inversion of configuration, from one molecule of 1 to the *N*-oxide of a second molecule, to form the anion of 2-hydroxypyridine-*N*-oxide (3) and a 1,2-dialkoxypyridinium cation (4). In the second step, the nature of which depends upon the alkyl group and the solvent, one of the alkyl groups of 4 is transferred, also with inversion of configuration, to one of the oxygens of 3. The product is then formed, without

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S.Wolfe,¹ Y.-H. Hsieh, R.J. Batchelor, F.W.B. Einstein, and I.D. Gay. Department of Chemistry, Simon Fraser University, Burnaby, BC V5A 1S6, Canada.

¹Corresponding author (telephone: (604) 291-5972; fax: (604) 291-5973; e-mail: swolfe@sfu.ca).

crossover and with net retention of configuration, by a double inversion of one alkyl group, or with crossover and net inversion of configuration, by single inversions of two alkyl groups.

Although four channels are, in principle, possible for the second step, only two have so far been observed. Based on kinetic studies, crossover experiments and measurement of the secondary H/D isotope effect, in dimethylformamide (DMF) at 140°C 1 (R = methyl) reacts exclusively via channel A. In the case of 1 (R = benzyl), crossover experiments and kinetic and stereochemical studies showed that channels A and B compete in DMF at 140°C, with channel A comprising about 80% of the reaction. In chloroform at 140°C, channels A and B compete equally.

Ollis and co-workers (5) have made the interesting observation that **5** undergoes quantitative Tieckelmann rearrangement to **6** in the solid state during 13 months at -15° C. The topochemical nature (6) of this reaction was not examined, but there are numerous related examples of intermolecular methyl transfer which proceed under topochemical control in the solid state (7). In each case, the crystal structure exhibits an intermolecular X···CH3···Y angle close to linearity



and an $X \cdots Y$ distance of 4 to 5 Å, and the reaction occurs exclusively, or is much faster, in the solid state than in solution or in the melt.

There is also an example of an intermolecular solid state rearrangement that is observable within 1 week at 40°C, but which, because of the molecular packing in the crystal, cannot proceed under topochemical control (8). This is the celebrated reaction $7 \rightarrow 8$ which, as a paradigm of linear methyl transfer (9), had previously been found to be intermolecular in solution (10). To account for the non-topochemical nature of the solid state reaction, Venugopalan et al. (8) have suggested that intermolecular methyl transfer can take place at defects such as microcavities, surfaces, and other irregularities in the ordered crystal arrangement.



2-Methoxypyridine-1-oxide (1, R = methyl) is reported to crystallize in two forms (11): anhydrous needles, mp 78 to 79°C, and hydrated prisms, mp 66.5 to 67.5°C. In our hands, despite considerable effort, only a hydrate, mp 73–76°C, was observed (1), as needles from ethyl acetate, and as tabular prisms after boiling in toluene, followed by cooling. The

tabular prisms were employed for the determination of the crystal structure. Figure 2 shows a portion of this crystal structure, including relevant geometrical parameters. The molecules in the set that includes **A**, **B**, and **C** are mutually related by simple translations in the *x*-direction, as are the molecules of the crystallographically distinct set that includes **D** and **E**. The figure displays an edge-viewed cross section of a sheet structure, two-dimensionally extended parallel to the *xy*-plane, which is the result of the formation of chains cross-linked by hydrogen bonds from water molecules to nitroxide oxygen atoms.

Since the methyl group is positioned anti to the C-N bond,² Fig. 2 strongly suggests that an intramolecular transfer of this group in the solid state is improbable. However, there are several potential pathways for an intermolecular, topochemically controlled Tiekelmann rearrangement: for example, a transfer of methyl groups from A to B and from **B** to **C** would convert **B** to product. Alternatively, a transfer of methyl groups from A to D and from D to B, or from A to **D** and **D** to **E**, etc., would convert **D** to product. The relevant O…O distances in the crystal, which range from 4.322(2)-4.451(2) Å, are close to the calculated gas phase value of 4.32 Å for the bimolecular complex, shown in Fig. 5 of ref. 1; however, the O…C…O angles in the crystal, which range from $107.5(2)-121.6(2)^{\circ}$, deviate substantially from linearity and from the calculated gas phase angle of 174.2°, also shown in Fig. 5 of ref. 1. This is one possible reason for our inability to observe an intermolecular solidstate reaction (9). A second is that, in the solid, each nitroxide oxygen is hydrogen-bonded to one or two water molecules, and it is known that the solvation of a nucleophile by just one water molecule greatly reduces the rate of an intermolecular gas-phase $S_N 2$ reaction (12).

A distinction between these possibilities became possible when one of us (Y.H.) discovered that a sample of crystalline 2-benzyloxypyridine-1-oxide (1, R = benzyl), which had been maintained at room temperature for some time, had undergone partial rearrangement to 2 (R = benzyl).

Figure 3 shows a portion of the crystal structure of 1 (R = benzyl).

It can be seen that, except for the absence of water molecules and the substitution of a phenyl group for one of the methyl hydrogens, there is a similar arrangement of the mol-

² The N-C-O-C torsion angles are $178.9(2)^{\circ}$ and $-178.5(3)^{\circ}$.

Fig. 2. A portion of the crystal structure of $\mathbf{1}$ (R = methyl), showing intermolecular O···O distances and O···C···O angles.



ecules of the methyl and benzyl compounds. Consequently, the possibilities for intermolecular topochemical rearrangement are also similar.

Referring to Fig. 3, molecules A, D, B, E, and C are sequentially related by a 2_1 -screw operation. There is a second crystallographically unique molecule in the structure which shows a closely similar arrangement about an alternate screw axis. One can visualize numerous combinations of relatively small rotations of molecules and (or) internal rotations about CH2-O, CH2-C, or Cring-O bonds which would produce the 180° angle required for the concerted transfer of a benzyl group, with inversion of configuration. For example: (*i*) transfer from **A** to **B** and **B** to either **C** or **E**, or from A to D and D to either B or E could produce 2 (R =benzyl) with net inversion of configuration in the benzyl group; alternatively, (ii) transfer from A to D, with inversion of configuration, followed by counterclockwise rotation of A, and return of the benzyl group to A with inversion of configuration will produce 2 with net retention of configuration in the benzyl group.

Figure 4 shows a portion of the crystal structure of 2 (R = benzyl).

Molecules, related by simple translations in *x*, are shown, and the nearest approaches between neighbouring NO and methylene groups are indicated. The N-O-C-C dihedral angle is $175.4(5)^{\circ}$. It is noteworthy that the much smaller intermolecular C-C···O-N torsion angles listed in Fig. 3 permit neither (*i*) nor (*ii*) above to produce this conformation initially, so that subsequent conformational rearrangement of **2** (R = benzyl) within the host crystal of **1** (R = benzyl) may be expected to lead to degradation of the crystal as the solid-state reaction proceeds.

The foregoing analyses of the two crystal structures permit the following tentative predictions: (*i*) the initial step of a solid-state Tieckelmann rearrangement of $\mathbf{1}$ (R = methyl, benzyl) will be slow, because of the substantial deviation of the entering and leaving groups from linearity; (*ii*) in the case of $\mathbf{1}$ (R = methyl), rearrangement is slowed further by **Fig. 3.** The crystal structure of $\mathbf{1}$ (R = benzyl), showing intermolecular O···O distances and O···C···O angles.



Fig. 4. The crystal structure of 2 (R = benzyl).



the presence of the water molecules;³ (*iii*) depending on the nature of the intramolecular and intermolecular librations in the crystal, the solid-state rearrangement of 1 (R = benzyl) could potentially produce 2 with net inversion of configuration, or with net retention of configuration; (*iv*) since the antiperiplanar N-O-C-C conformation of 2 (R = benzyl) can probably not be accommodated within the host lattice of 1 (R = benzyl), the reaction likely proceeds at domain boundaries or packing defects and will be accompanied by a loss

³ The intermolecular RCH₂...O distances are from 0.15–0.36 Å larger in 1 (R = methyl, hydrate) than in 1 (R = benzyl).

200

Fig. 5. (*a*) The solid-state ¹³C NMR spectrum of a fresh sample of **1** (\mathbf{R} = benzyl); (*b*) the spectrum after 10 months; (*c*) the spectrum after 25 months; (*d*) the spectrum after 36 months; (*e*) the spectrum of an authentic sample of **2** (\mathbf{R} = benzyl).



100

ppm

٥

The conversion of 1 to 2 (R = benzyl) was also followed at room temperature by solid-state ¹³C NMR, and was also found to occur on a time scale of months. Figure 5 shows: (a) the spectrum of a fresh sample of 1; (b) the spectrum after 10 months; (c) the spectrum after 25 months; (d) the spectrum after 36 months; (e) the spectrum of an authentic sample of **2**. While not all carbon resonances are resolved in the solid, the occurrence of the reaction is easily seen from the decay of the peaks at 70, 110, and 118 ppm, and the growth of peaks at 79, 106, 132, and 135 ppm. There is a steady conversion of 1 to 2, and no peaks corresponding to intermediates are observed. Unfortunately, 1 has a long spinlattice relaxation time, necessitating a repolarization delay of 90 s. This makes it impractical to signal average to a significantly better signal-to-noise ratio than shown. Consequently the presence of intermediates at levels up to a few percent of the total cannot be excluded.

Assuming that the rearrangement occurs in more than one step, with each step slow for the reasons already stated, transient formation of 3 and 4 can be expected, but these would only have been observable in the initial stages of the reaction.

To determine the stereochemical course of the rearrangement, a sample of optically active 2-(α -deuteriobenzyloxy)pyridine-1-oxide was synthesized as described previously (1, 13), carefully crystallized from ethyl acetate, and allowed to stand for 11 months. At this time the rearrangement was found to be 85% complete. The reactant and product were separated, benzyl alcohol was released from each, by treatment with sodium methoxide in the case of **1**, and by treatment with zinc and acetic acid in the case of **2**, and the alcohols were converted to their Mosher esters (1, 14) for determination of their ¹H NMR spectra.

Figure 6 shows the deuterium-decoupled ¹H NMR spectra of the benzyl protons of the Mosher esters of: (A) the

Fig. 6. Deuterium-decoupled ¹H NMR spectra of the benzyl protons of the Mosher esters of: (*a*) chirally labelled benzyl- α -D-al-cohol; (*b*) chirally labelled benzyl- α -D-alcohol recovered from **1** (R = benzyl), after partial rearrangement to **2**; (*c*) chirally labelled benzyl- α -D-alcohol recovered from **2** after partial rearrangement.



benzyl- α -d-alcohol employed to prepare the initial sample of **1**; (B) the benzyl- α -D-alcohol recovered from **1** following rearrangement; (C) the benzyl- α -D-alcohol recovered from **2** following rearrangement. Samples (A) and (B) are identical; sample (C) is the epimer of (A).

It follows that the solid-state rearrangement of 1 (R = benzyl) proceeds with essentially complete inversion of configuration, in contrast to the 50–80% inversion observed in

solution. Based on our earlier examination of Fig. 3, it can be concluded that the topochemical rearrangement is an intermolecular process that proceeds by transfer of a benzyl group from A to B and B to C, etc., or by transfer from A to **D**, **D** to **B**, **B** to **E**, and **E** to **C**, etc., with inversion of configuration in every step.

Experimental

NMR Studies

The sample used for NMR experiments was supplied as a powder of fine crystals. This was loosely packed into a segment of 5 mm NMR tubing, and remained in this tube over the course of all measurements. The sample had a tendency to "clump" over time, and was occasionally stirred with a spatula and repacked. During the whole course of the NMR experiments, the sample was exposed to the atmosphere, and was maintained at ambient laboratory temperature, 23 \pm 5°C. Probe temperatures during NMR experiments were also within this range.

Solid-state ¹³C NMR spectra were recorded at 37.56 MHz on a 3.5T instrument. ¹H-¹³C cross-polarization was used, with a contact time of 2 ms and a relaxation delay of 90 s. The sample was spun at the magic angle at 2.4 kHz; at this speed and field, small spinning sidebands are seen for the sp carbons. These were suppressed, in the spectra shown, by a phase-cycled TOSS pulse sequence. Proton decoupling fields of 50-85 kHz were used, the higher fields giving a slight improvement in line width. Resonances were referred to TMS using solid adamantane as a secondary standard, taking the shift of the high-frequency line to be 38.56 ppm.

Stereochemical studies

Full details of the synthesis of chirally deuterated 1, of the recovery of benzyl- α -D-alcohol from 1 and 2, of the conversion of these alcohols to Mosher esters, and of the NMR analyses of these esters can be found in ref. 1.

X-ray crystallography

One large crystal of 1 (hydrate, $R = methyl)^4$ (obtained from toluene solution and believed to be anhydrous) was exposed to a dry nitrogen atmosphere in a crystallographic glove box while it was cleaved to produce the fragment used for crystallographic analysis. When the crystal was exposed to the dry atmosphere it appeared to begin to liquefy. Nonetheless a fragment was successfully sealed in a capillary tube, after which time it ceased to "melt" while the remaining fragments liquefied completely. It seems likely that the sample was undergoing dehydration in the glove box to produce a lower melting substance or mixture. Full-matrix least-square refinement, on F, of 254 parameters for 1558 data $(I_0 > 2.5 \sigma(I_0))$ included independent coordinates for all atoms, anisotropic displacement parameters for the nonhydrogen atoms, and a single isotropic thermal parameter for each of the following three groups of hydrogen atoms: those on the C₅N-rings, those of the methyl groups, and those of the water molecules.

A crystal of 1 (R = benzyl)⁵ obtained from ethyl acetate solution was mounted on a glass fiber using epoxy adhesive. While less than 1% decays of the intensity standards were observed during data acquisition, remeasurement of three of the stronger reflections showed a systematic decay over a period of 130 days, by which time the net intensities had all dropped to 28% of their original values. This net loss of intensity appeared to apply equally to all the reflections tested. A peak search at the end of that time revealed only reflections indexed on the original cell and orientation. Fullmatrix least-squares refinement, on F, of 273 parameters for 1728 data $(I_0 > 2.5\sigma(I_0))$ included independent coordinates and anisotropic displacement parameters for the nonhydrogen atoms. Hydrogen atoms were riding in calculated positions (C—H, 0.95 Å) and were initially assigned isotropic thermal parameters proportionate to the equivalent isotropic parameter of the respective carbon atoms. Subsequently, all hydrogen atom isotropic thermal parameters were constrained to have equivalent shifts.

A fragment cleaved from a crystal of 2 (R = benzyl)⁶ obtained from hexane solution was mounted on a glass fiber using epoxy adhesive. Full-matrix least-squares refinement, on F, of 138 parameters for 570 data $(I_0 > 2.5\sigma(I_0))$ was conducted analogously to that of 1 (R = benzyl).

All data were acquired at room temperature using an Enraf-Nonius CAD-4F diffractometer and with graphitemonochromatized Mo K_{α} radiation. Complex scattering factors for neutral atoms (15) were used in the calculation of structure factors. The programs used for data reduction, structure solution, and graphical output were from the NRCVAX Crystal Structure System (16). The program suite CRYSTALS (17) was employed in the refinement. Full details of the X-ray crystallographic structure determinations have been deposited as hardcopy and in CIF format.⁷

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⁴Crystal structure of **1-Me-hydrate**: colourless prism, C₆H₇NO₂·1.5H₂O, triclinic, space group $P\overline{1}$, Z = 4, a = 6.5109(8) Å, b = 6.8620(13) Å, c = 17.4633(23) Å, $\alpha = 79.511(13)^\circ$, $\beta = 88.944(11)^\circ$, $\gamma = 73.374(12)^\circ$, V = 734.64 Å³, T = 293 K, $R_F = 0.028$, GoF = 1.49. ⁵Crystal structure of **1-Bz**: colourless prism, $C_{12}H_{11}NO_2$, monoclinic, space group $P2_1/a$, Z = 8, a = 16.4480(20) Å, b = 6.3356(7) Å, c = 20.011(2) Å 20.941(3) Å, $\beta = 111.597(9)^{\circ}$, V = 2029.0 Å³, T = 293 K, $R_{\rm F} = 0.041$, GoF = 1.86. ⁶ Crystal structure of **2-Bz**: colourless prism, $C_{12}H_{11}NO_2$, orthorhombic, space group $Pc2_1$ b,; Z = 4, a = 5.8898(5) Å, b = 13.0782(14) Å, c = 12.0782(14) Å, c

^{13.3848(12)} Å, v = 1031.00 Å³, T = 293 K, $R_{\rm F}$ = 0.040, GoF = 1.59.

⁷ Full details of the crystallographic structure determinations, displacement ellipsoid plots of the molecular structures, and a diagram of the extended H-bonding in 1-Me-hydrate, tables of crystallographic details, atomic coordinates, distances and angles, anisotropic displacement parameters, and least-squares planes are available as supplementary data, and may be purchased from the Depository of Unpublished Data, Documentary Delivery, CISTI, National Research Council Canada, Ottawa, ON K1A 0S2, Canada (http://www.nrc.ca/cisti/irm/unpub_e.shtml for information on ordering electronically). The CIF file is also available. Tables of crystallographic details, atomic coordinates, and distances and angles have also been deposited with the Cambridge Crystallographic Data Centre. Copies of the data can be obtained free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. (Fax: 44-1223-336033 or e-mail: Structure factors are no longer being deposited and may be obtained from the author.

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