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Trifluoromethylation of pyridines through skeletal rearrangement

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We report a skeletal rearrangement of pyridines that enables functionalisation of the 2 and 3-positions, *via* Zincke ketone intermediates. The ring-opened pyridines undergo selective reaction with cheap and readily available acylating agents, setting up an alternative ring closure pathway to afford rearranged structures incorporating groups such as CF_3, CF_2H, and CO_2Et at the pyridine C2 position. The process enables highly selective C2 over C4 trifluoromethylation of pyridines, a reaction that challenges conventional Minisci methods. Sequential functionalisation of the Zincke intermediate is further demonstrated, to access triply-substituted pyridine products from mono-substituted starting materials.

Introduction

Heteroarene ring-opening/ring-closing rearrangements have a rich history in synthesis, enabling skeletal re-organisations to be exploited as strategic disconnections. The Dimroth rearrangement from 1909 is archetypal (Scheme 1A),[1] first observed on amino-triazoles, and since expanded to numerous nitrogenated aromatic systems. General features consist of an initial ring-opening, typically *via* pericyclic reaction or nucleophilic addition, to an open-chain intermediate (*e.g.* **2**). A σ-bond rotation then enables ring closure *via* an alternative atom, with a resultant switching of substituent positions. For the Dimroth rearrangement, the heteroarene undergoes overall single-atom exchange (N to N), illustrated in Scheme 1B for a process-scale synthesis of a drug candidate developed by Goundry and co-workers.[2] Other well-known examples include the Cornforth rearrangement of acyloxazoles,[3] a thermal pericyclic reaction proceeding through a nitrile-ylide intermediate that leads to a double atom switch (C and O, elegantly applied to macrocyclic peptide contraction by the Yudin lab, Scheme 1C),[4] and the Kost–Sagitullin pyridine recyclisation to give arene products[5] (N=C switching for C=C in this recent example from Kano, Morofuji and co-workers, Scheme 1D).[6]

The Zincke reaction of pyridiniums is a classic heteroarene ring-opening/ring-closing reaction, but it does not typically lead to skeletal rearrangement (Scheme 1E). Rather, the original reaction enables exchange of the NR moiety, whereby the open-chain Zincke intermediate **12** undergoes amine exchange and re-closure to preserve the initial pyridine skeleton connectivity.[7] Recent advances in Zincke chemistry have shown it is possible to functionalise the open-chain intermediate **12** in new ways, which on re-closure give valuable 3- and 4-substituted pyridine derivatives **13** that are difficult to obtain directly.[8–16] This approach has been extended to several recent skeletal-editing studies of pyridines, where the addition of nucleophile ring-opening/ring-closing (ANRORC) mechanism sets up a transformation into a different heterocycle.[17–20] Our work in this area[9,14,18] prompted us to consider a functionalisation – rearrangement mode, whereby a pyridine is opened to the Zincke intermediate **15**, and functionalised such that an alternative closure pathway is created. Such a rearrangement could create new ways of installing pyridine functionality *via* skeletal reorganisation.[21]

The idea is set out in Scheme 1E, showing an electrophilic acylating agent such as TFAA reacting regioselectively with the Zincke imine **15**. This could then set up an alternative ring-closing pathway, *via* the newly introduced trifluoroacyl carbonyl group. Under the ammonia ring-closure conditions typical for pyridine reformation, preferential imine formation could take place at the newly introduced ketone group to give the skeletally re-organised pyridine **17**. The original C2 atom is now external to the ring at the C3 position, and the acylating agent has introduced a new CF_3 group at C2. Direct trifluoromethylation at the pyridine C2 position has received considerable attention in the literature, given the relevance of both azines and the CF_3 group to drug design.[22] The proposed route would offer a novel, economic alternative to Minisci-based and related methods, which often employ expensive sources of CF_3 and struggle to discriminate between the C2 and C4 positions.

Results and discussion

We began by examining trifluoromethylation of the Zincke imine **15a** derived from 2-phenyl pyridine **14a**, using trifluoroacetic anhydride (TFAA) (Table 1). Acylation of Zincke

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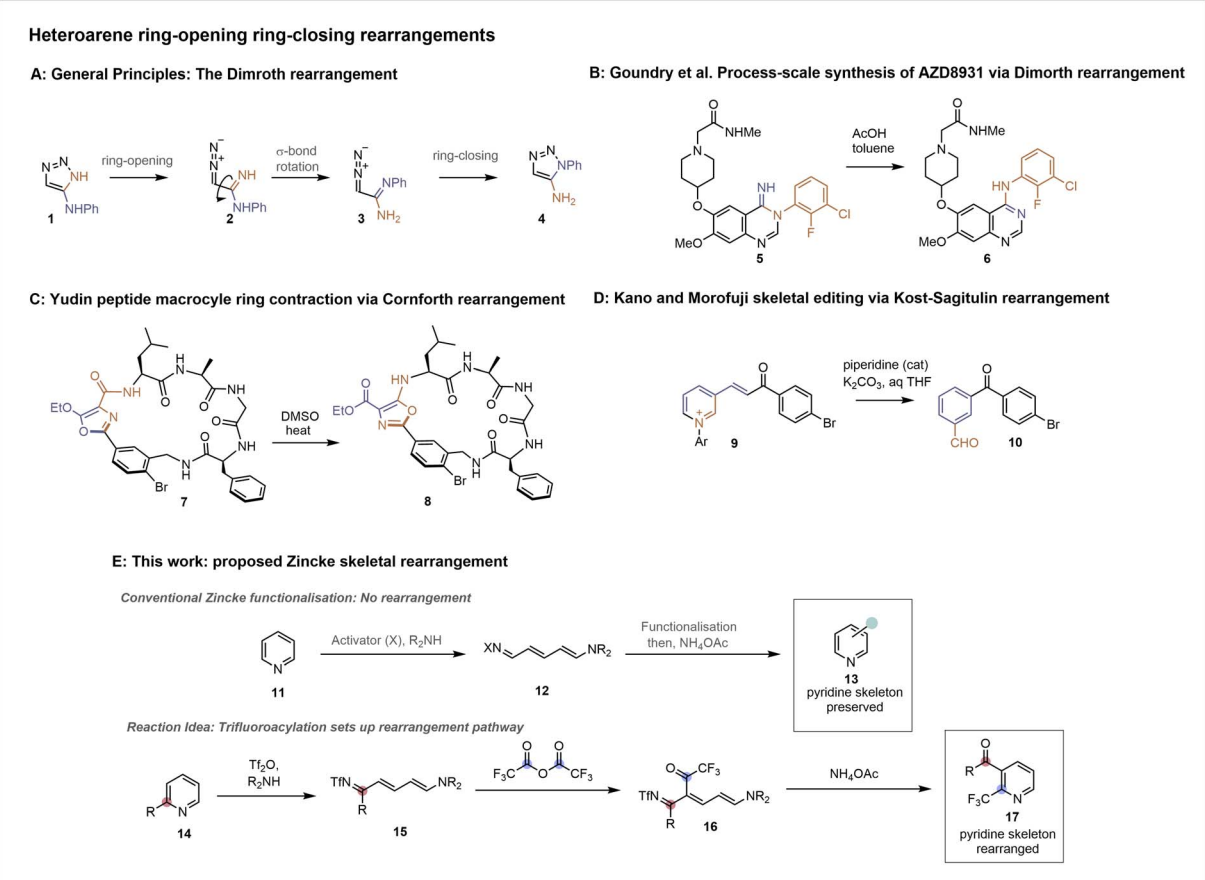
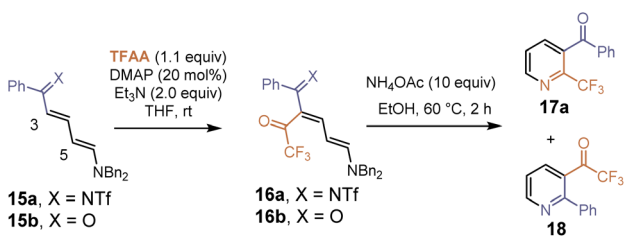


Scheme 1 (A–D) Heteroarene ring-closing/ring-opening rearrangement. (E) Zincke imine reaction and proposed Zincke skeletal rearrangement idea.

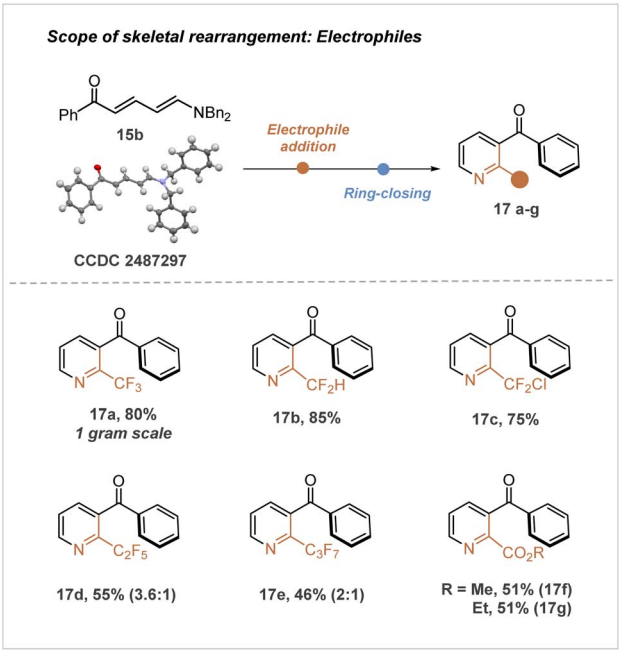
Table 1 Reaction development



Entry	Starting material	Variation	Yield of **17a** (%)
1	**15a**	None	<10
2	**15b**	None	80
3	**15b**	Without DMAP	0
4	**15b**	60 °C in step 1	77
5	**15b**	0 °C in step 1	80
6	**15b**	Overnight step 1	80

imines has yet to be described in the literature, although two methods for Vilsmeier formylation were recently disclosed: The Fu lab demonstrated formylation of related streptocyanines derived from 3- and 4-substituted pyridines, and the Gryko lab reported formylation of 2-arylpyridines *via* Zincke imines to access rearranged ketone products.[15,23,24] Our initial trials with TFAA were low-yielding, but instructive; we were pleased to observe the desired trifluoroacetylated product **16a**, but only at low conversions. Further, when **16a** was heated with NH_4OAc, we observed equal amounts of both pyridine regioisomers **17a** (rearranged) and **18**. Electrophilic addition had taken place exclusively at the 3-position, essential for the postulated reaction to work, but the presence of the NTf imine appeared problematic in terms of both ring-closure and overall efficiency of the acylation. Switching to the Zincke ketone **15b**, available through simple hydrolysis, solved both problems. When **15b** was treated with TFAA in THF at room temperature, followed by heating with NH_4OAc in EtOH, we were delighted to observe the formation of the rearranged 2-trifluoromethylpyridine **17a** in a very good 80% yield. A short optimisation study of temperature and reaction time led to no further improvements (Table 1), so we took these conditions forward to investigate reaction scope.

Initially, we were interested in examining the scope of acylating agents that could react with the Zincke ketone to access differentially functionalised pyridines at C2 (Scheme 2). We were pleased to see that both the electrophilic reagents $(F_2HCCO)_2O$ and $(F_2ClCCO)_2O$ were successful, delivering the

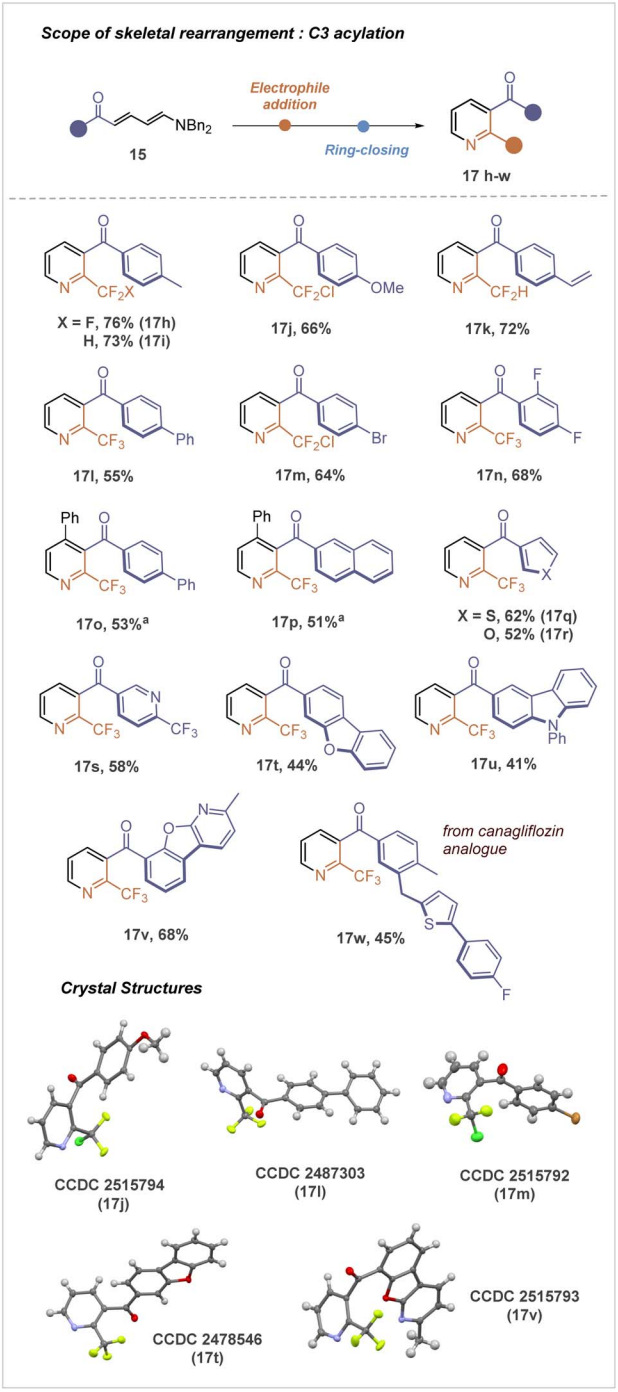


Scheme 2 Substrate scope. Conditions: (A) electrophile addition step: Zincke ketone (0.2 mmol), acylating agent (1.1 equiv.), DMAP (20 mol%), triethylamine (2 equiv.), THF (0.1 M), r.t., 2 h. (B) Ring-closing step: NH_4OAc (10 equiv.), EtOH (0.05 M), 60 °C, 2–4 h.

CF_2H and CF_2Cl substituted pyridines **17b** and **17c** in good yield. Both the CF_2H and CF_2Cl groups are valued in medicinal and agrochemistry for their lipophilic H-bonding and halogen-bonding properties, respectively,[25] and are typically introduced *via* Minisci radical reactions. While these methods are well developed for the generation of the respective radicals,[26] selectivity for the 2 over the 4-position is not generally possible, and mixtures of pyridines are generated.

We could also introduce the perfluoro congeners C_2F_5 and C_3F_7 with a similarly effective acylation step, but lower regioselectivity in the pyridine recyclisation (overall yields of 55% and 46%). It appears that the greater steric bulk of the perfluoroalkyl groups diminishes the selectivity between ketone groups in the ammonia cyclisation step. We were also successful with oxalyl reagents to access the picolinate derivatives, **17f** and **17g**, introducing ester functionality to complement the perfluoroalkyl building blocks. The conditions were effectively scaled up to gram scale for Zincke ketone **15b** (3 mmol), delivering the rearranged product **17a** in an identical 80% yield. Less electrophilic acyl donors were ineffective in the reaction, with simple Ac_2O, AcCl and BzCl undergoing no reaction with the Zincke ketone.

With the scope of acylation and the C2 substituent established, we moved on to examine the C3 aroyl group installed through the rearrangement (Scheme 3). We used trifluoroacetic anhydrides, difluoroacetic anhydrides, and chlorodifluoroacetic anhydrides as the model electrophilic agents, with the Zincke ketones derived from 2-aryl substituted pyridines. The reaction was generally tolerant of various aryl functionality; Zincke ketones with an electron-neutral and electron-donating phenyl substituent worked effectively (**17h**, **17i** and **17j**), along with extended conjugated substituents such as vinyl (**17k**) and phenyl groups (**17l**). The structure of this biphenyl derivative was confirmed by X-ray crystallography (CCDC 2487303), illustrating the CF_3 substituent forcing the pyridyl group to twist out of planarity with the 3-aroyl substituent.



Scheme 3 Substrate scope. Conditions: (A) electrophile addition step: Zincke ketone (0.2 mmol), acylating agent (1.1 equiv.), DMAP (20 mol%), triethylamine (2 equiv.), THF (0.1 M), r.t., 2 h. (B) Ring-closing step: NH_4OAc (10 equiv.), EtOH (0.05 M), 60 °C, 2–4 h. [a] Trifluoroacetylation step was performed at 0 °C.

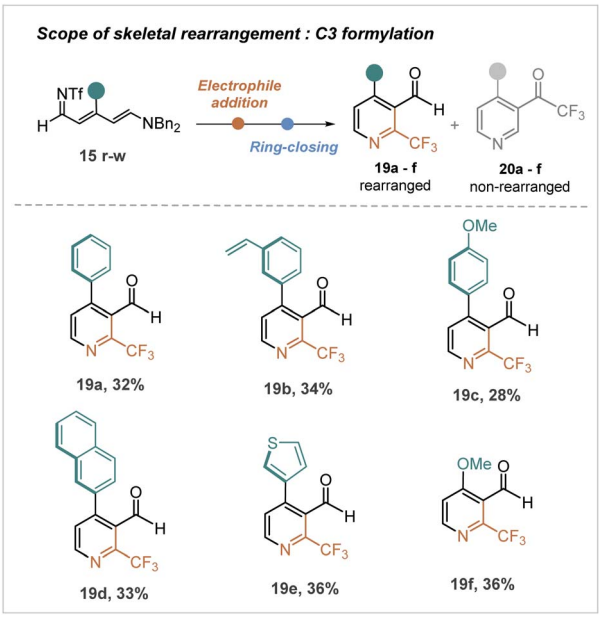
Halogenated, disubstituted, and electron-withdrawing Zincke ketones were likewise found to undergo rearrangement in a good yield (**17m** and **17n**). Zincke ketones derived from 2,4-disubstituted pyridines were also found to react effectively with TFAA to furnish more densely functionalised pyridine products (**17o** and **17p**); however, Zincke ketones derived from 2,5-disubstituted pyridines were not viable in the TFAA reaction. Next, we synthesised a variety of heteroaryl-substituted Zincke ketones to study the C3 acylation with trifluoroacetic anhydride. Thiophene and furan-substituted Zincke ketones were good substrates (**17q** and **17r**), along with the Zincke ketone derived from a substituted bipyridine to give the pseudo-symmetric C3 acylated compound (**17s**). Increasing the complexity, we used tricyclic (hetero)aromatic oxacycle and azine, such as dibenzofuran-, carbazole- and benzofuropyridine-substituted Zincke ketones, with trifluoroacetic anhydride.

We were pleased to see that each reacted well with TFAA, delivering C3 acylated heterocycles (**17t**, **17u**, and **17v**). These groups are valuable in medicinal and material chemistry due to their extended conjugation and photophysical properties. The structures of these valuable compounds were confirmed by X-ray crystallography (CCDC 2478546 and CCDC 2515793). We were also able to derivatise the aryl motif of the diabetes medication canagliflozin, giving **17w** in 45% yield. Zincke ketones from simple 2-alkyl-substituted pyridines did not react with TFAA (see SI for a list of challenging substrates).

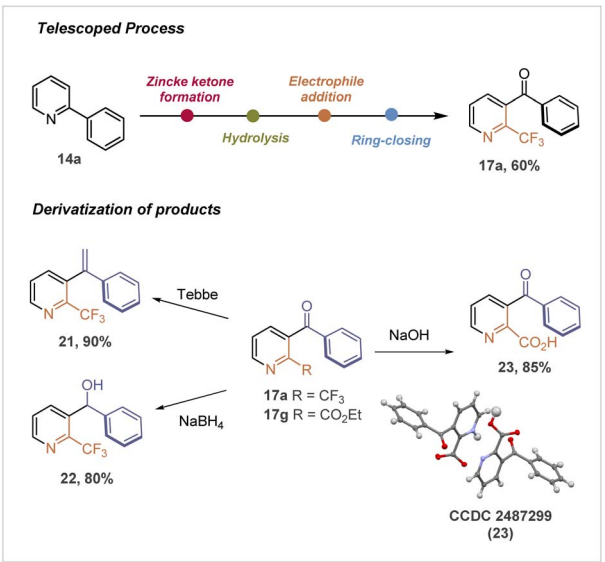
We next moved on to investigate the viability of *para*-substituted pyridines (Scheme 4). Trifluoroacylation was feasible on the Zincke aldimine derivative **15** directly, with ring closure giving a *ca.* 1 : 2 mixture in favour of the non-rearranged product **20a**, representing direct *meta*-trifluoroacylation. As expected, the electrophilic aldimine is competitive with the trifluoroacyl group in the ring-closure step, leading to both constitutional isomers. We could isolate the skeletal-rearranged products **19a–19f** as the minor components, incorporating a trifluoromethyl substituent at C2 and with the original C2 carbon now rearranged into the 3-formyl group. The formyl group presents a versatile vector for further functionalisation, complimenting the aroyl group installed from 2-substituted pyridines.

The sequence could be conducted as a telescoped process with no chromatographic purification of intermediates, starting from 2-phenyl pyridine **14a** (Scheme 5). The Zincke ketone was made in a one-pot procedure, followed by simple work-up and concentration. The crude material was then subjected to trifluoroacetylation and NH_4OAc treatment to give the final pyridine product **17a** in 60% overall yield. The reaction transfers an aryl group from the pyridine 2-position, a very accessible position to functionalise with numerous analogs available commercially, to the more challenging 3-position as an aroyl unit. We could further demonstrate the derivatizations of this 3-aroyl group to access diarylmethane derivatives, versatile building blocks with widespread application in chemistry and materials. Methylenation with Tebbe's reagent gave the 1,1-diarylethylene **21**, and we could reduce the ketone with $NaBH_4$ in butanol to access the diarylmethanol **22**. We synthesised 3-benzoyl picolinic acid **23** by alkaline hydrolysis of our rearrangement picolinate product **17g**, with X-ray analysis of the α-iminocarboxylic acid structure showing a unit cell consisting of two molecules – one neutral and the other in zwitterionic form.

The nucleophilicity of the Zincke intermediate enables functionalisations that would not be possible directly on the pyridine. We wondered if we could further expand the viability of our skeletal rearrangement *via* sequential electrophilic addition, to achieve two C–H functionalisations. The Zincke



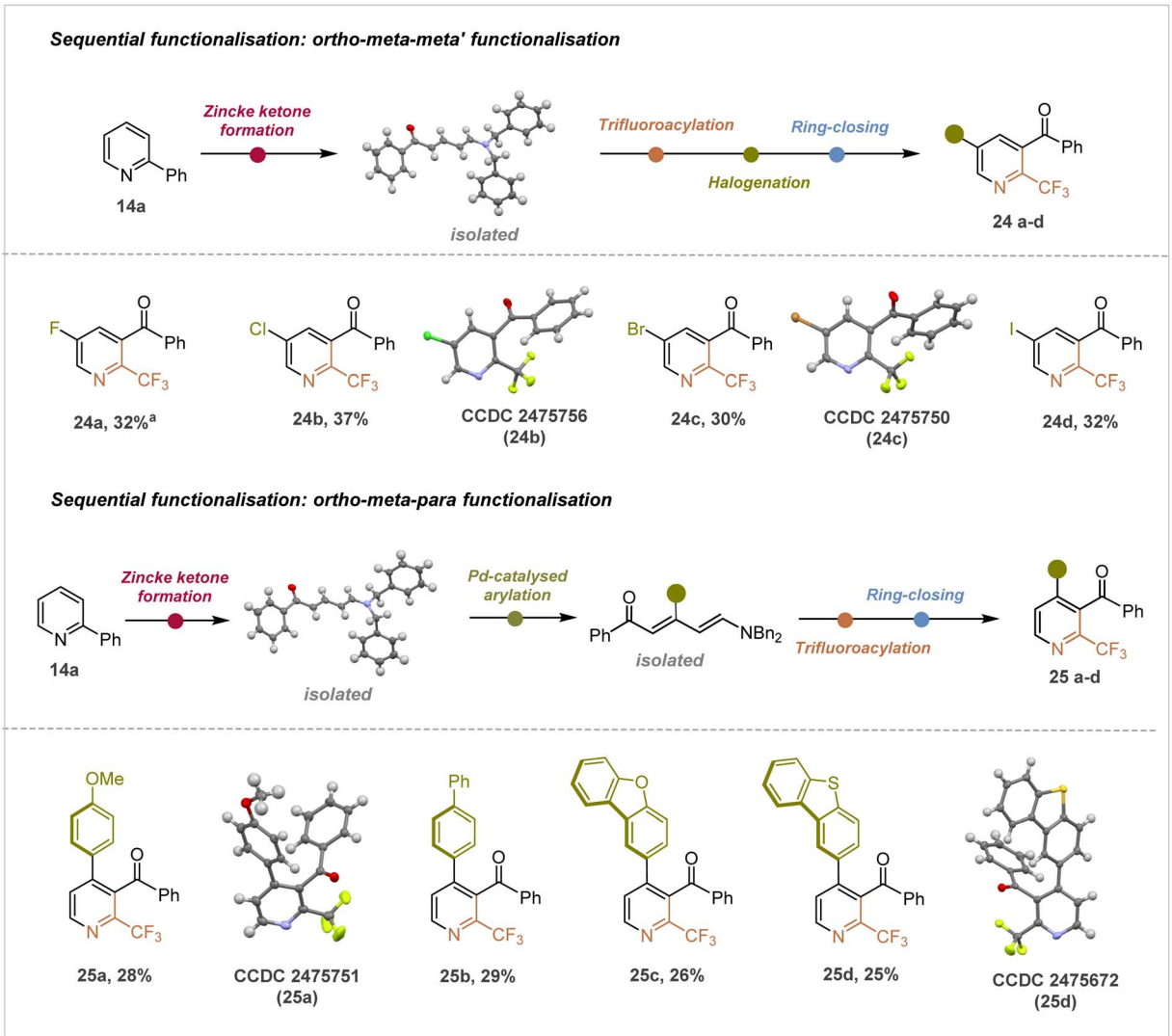
Scheme 4 Substrate scope. Conditions: (A) electrophile addition step: Zincke imine (0.2 mmol), trifluoroacetic anhydride (1.2 equiv.), DMAP (20 mol%), triethylamine (2 equiv.), THF (0.1 M), 40 °C, 2 h. (B) Ring-closing step: NH_4OAc (10 equiv.), EtOH (0.05 M), 60 °C, 2–4 h. *The rest of the mass balance corresponds to the non-rearranged product.



Scheme 5 Telescoped process and synthetic utility.

intermediate is nucleophilic at both C3 and C5, with preference for C3 generally being observed for simple electrophiles. An initial C3-selective trifluoroacetylation followed by the addition of a second electrophile could access sequentially *meta*-substituted pyridines, following ring closure with NH_4OAc. We investigated this idea through trifluoromethylation of Zincke ketone derived from 2-phenylpyridine **14a** at C3 to set up the skeletal rearrangement, followed by treatment with either NFSI, NCS, NBS, or NIS as described by McNally.[8,10] Ring closure in the usual way then gave each of the 2-trifluoromethyl-3-aroyl-5-halo pyridines (**24a–d**) in good overall yield, with the sequential Zincke ketone functionalisation taking place in a one-pot, telescoped process (Scheme 6).

We were interested in extending this sequential pyridine functionalisation to C–C bond formation, using our previously developed C4-selective Heck-arylation method.[9] We found that the trifluoroacetylated Zincke ketone **16b** (Table 1) performed poorly under the Pd-catalysed C4 arylation conditions, with the trifluoroacetyl group hindering the Heck arylation step. Altering the reaction sequence to begin with the Heck reaction, however, was productive (Scheme 6), with the arylated Zincke ketone being purified by chromatography. Subsequent trifluoroacetylation tolerated the flanking aryl substituent at C4, enabling *in situ* ring closure with NH_4OAc to access the 2,3,4-trisubstituted pyridines containing both aryl and heteroaryl groups (**25a–d**). X-Ray analysis of **25a** and **25d** confirmed the



Scheme 6 Polyfunctional pyridine synthesis with overall yields from 2-phenylpyridine. Conditions for halogenation-trifluoromethylation: (A) Zincke ketone formation: 2-phenylpyridine (0.2 mmol), Tf_2O (1.0 equiv.), EtOAc (0.1 M), −78 °C, 30 min, then Bn_2NH (1.2 equiv.), collidine (1.0 equiv.), 30 min, concentrate. Then K_2CO_3 (2.0 equiv.), THF : H_2O (4/1, 0.1 M), 100 °C, 18–24 h. (B) Trifluoroacylation: Zincke ketone (0.2 mmol), trifluoroacetic anhydride (1.1 equiv.), DMAP (20 mol%), triethylamine (2 equiv.), THF (0.1 M), r.t., 2 h. (C) Halogenation step: *N*-halosuccinamide (1.1 equiv.), r.t., 1 h. (D) Ring-closing step: NH_4OAc (10 equiv.), EtOH (0.05 M), 60 °C, 2–4 h. [a]NFSI (1 equiv.) was used, stirred at r.t. overnight. Conditions for arylation-trifluoromethylation: conditions as previous for Zincke ketone formation, trifluoroacylation (performed at 0 °C), and ring closing. Arylation step: Zincke ketone (0.2 mmol), aryl halide (1.2 equiv.), $Pd(OAc)_2$ (10 mol%), K_2CO_3 (2 equiv.), THF (0.1 M), 100 °C, overnight, followed by work-up and chromatography.

structures and showed that the three arene units are successively orthogonal to one another, projecting substituents into distinct areas of structural space.

Conclusions

In summary, we have developed a novel pyridine ring-opening/ring-closing skeletal rearrangement that enables a number of C2-functionalisations. The process introduces electron-poor groups such as CF_3, CF_2H, and CO_2Et to the 2-position exclusively using cheap and readily available acylating agents, complementing unselective Minisci-type radical approaches. The facility of Zincke ketones to undergo sequential functionalisation enables both 2,3,5- and 2,3,4-tri-substituted pyridines to be synthesised from 2-substituted pyridine starting materials, for both halogenation and arylation reaction classes, generating versatile di(hetero)arylmethane products. Further applications of this skeletal rearrangement are underway in our laboratory.

Author contributions

H. W., N. B. and M. F. G. conceived and designed the experiments. H. W. and N. B. performed the experiments and analysed the data. H. W., N. B. and M. F. G. wrote the manuscript.

Conflicts of interest

There are no conflicts to declare.

Data availability

Details on the procedures and the corresponding datasets reported in this study are provided in the paper and its supplementary information (SI) files, and are available from the corresponding author on request. Supplementary information: all preparative procedures, characterisation data, and NMR spectra. See DOI: https://doi.org/10.1039/d6sc04438g.

CCDC 2487297, 2515794, 2487303, 2515794, 2478546, 2515793, 2487299, 2475756, 2475750, 2475751 and 2475672 contain the supplementary crystallographic data for this paper.[27a–j]

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