

README file

Dataset Title: **Dataset of “Electrochemical oxidative CF₃ radical-induced lactonization and etherification of terminal and internal alkenes”**

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Dataset contents

The dataset consists of:

- 1 compressed folder named **1H19F13C-NMR_Dataset.zip** containing 28 subfolders named
3a, 3b, 3c, 3d, 3e, 3f, 3g, 3h, 3i, 3j, 3k, 3l, 3m, 3n, 3o, 3p, 3q, 3s, 3t, 3u, 6a, 6b, 6c, 6d, 6e, 6f, 6g, 6h
each subfolder contains ¹H, ¹⁹F, ¹³C-NMR spectra files in. fid format.
- 1 folder in .pdf format named **1H19F13C-NMR_Scheme 1-2-3.pdf**
- 1 readme file in .pdf format named **1H19F13C-NMR_README.pdf**

Dataset documentation

Abstract

The dataset contains all the NMR spectra of the CF₃ containing lactones and cyclic ethers synthesized with an electrochemical strategy. The protocol developed facilitates the synthesis of functionalized lactones and cyclic ethers from both terminal and internal alkenes exploiting the advantages of electrochemistry. Mechanistic insights, supported by cyclic voltammetry, radical trapping experiments, and DFT calculations, confirm the involvement of CF₃ radicals. This metal-free protocol offers a sustainable and practical approach for accessing pharmacologically relevant scaffolds, underlining its potential for applications in medicinal chemistry.

Content of the files

The attached dataset includes the NMR spectra of all lactones and cyclic ethers (compounds **3a-3q**, **3s-3u** and **6a-6h**) presented in Scheme 1, Scheme 3 and Scheme 2, respectively, as shown in the 'Scheme 1,2,3' file included in the document

Methodologies

^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded on Varian 400-MR (400 MHz) (equipped with autoswitchable PFG probe) and Bruker Advance Neo 600 MHz (equipped with CryoProbe Prodigy Broadband 5mm) spectrometers. NMR multiplicities are abbreviated as follows: s = singlet, d = doublet, t = triplet, q = quartet, spt = septet, m = multiplet, bs = broad signal. Coupling constants J are given in Hz. All ^1H and ^{13}C chemical shifts are calibrated to residual protic-solvents, while ^{19}F chemical shifts were referenced to the internal standard trifluorotoluene.

Protocol

The ElectraSyn vial (10 mL), equipped with a magnetic stir bar, was charged with the electrolyte LiClO_4 (0.2 M), the alkene **1** or **5** (0.15 mmol, 1.0 equiv), and the Langlois reagent **2** (0.22 mmol, 1.5 equiv). The ElectraSyn vial cap, fitted with an anode and cathode, was secured to the vial and sealed with a rubber septum. The vessel was evacuated and backfilled with argon three times.

Dry ACN (6 mL) was then added together with the selected percentage of an acid, and the solution was degassed by bubbling with argon under stirring for 1 minute. The reaction mixture was electrolyzed for 3 hours. Upon completion, PhCF_3 (0.15 mmol) was added as an internal standard, and the yield was determined via ^{19}F NMR spectroscopy.

Afterward, the ElectraSyn vial cap was removed, and the electrodes and vial were rinsed with EtOAc (10 mL) and aqueous NaHCO_3 (10 mL). These rinses were combined with the crude reaction mixture in a separatory funnel. The organic layer was separated, and the aqueous layer was extracted with EtOAc (2 \times 10 mL). The combined organic layers were dried over Na_2SO_4 , concentrated under reduced pressure, and purified by flash chromatography (Hex:EtOAc from 95:5 to 7:3) to yield the pure products **3** or **6** (see Scheme 1,2,3 included in the document)

References

Riccardo Grigolato, Tommaso Fantoni, Giuseppe Autuori, Matteo Lattanzi, Lucia Ferrazzano, Walter Cabri, Alessandra Tolomelli, Electrochemical oxidative CF_3 radical-induced lactonization and etherification of terminal and internal alkenes. RSC Advances, 2025, Accepted.