Multicomponent Reaction for the Synthesis of Pyrido[1, 2-b] isoquinoline derivatives via [3+2] Cycloaddition Reaction between Alkynes and in situ Generated Isoquinolininium ylides

Sundar S. Shinde, a Soumi Laha, a Dharmendra K. Tiwari, b B. Sridhar, c and Pravin R. Likhar a

Catalysis & Fine Chemical Division, X-Ray Crystallography Centre, CSIR- Indian Institute of Chemical Technology, Uppal Road, Tarnaka Hyderabad-500007, INDIA, Molecular Synthesis and Drug Discovery Unit, Centre of Biomedical Research(CBMR), SGPGMIS Campus, Raebareli Road, Lucknow, 226014, UP, India.

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1. **General Information:**

All substrates and reagents were readily and commercially available. TLC analysis was performed using pre-coated glass plates. Column chromatography was conducted using silica gel (60-120 mesh). All $^1$H and $^{13}$C NMR spectra were recorded in deuterated chloroform (CDCl$_3$) on Avance 300 or 400 or Avance 500 spectrometers. Chemical shift ($\delta$) are reported in parts per million (ppm) relative to residual CHCl$_3$ ($^1$H: $\delta$ 7.26 (ppm), $^{13}$C: $\delta$ 77.00 (ppm) as an internal reference. Coupling constant ($j$) is reported in (Hz). Peak multiplicities are indicated as: s-singlet, t-triplet, q-quartet, m-multiplate and dd-doublet of doublet. Mass spectra were recorded by using 70 Ev spectrometer. High resolution mass spectrums (HRMS) were recorded using Applied Bio-sciences HRMS spectrometer at national center for mass spectroscopy.
Spectral copies of $^1$H NMR and $^{13}$C NMR
N\_O\_O\_C\_O\_Et (4m)
COSY for compound (4v):
NOESY for compound (4v):
HMBC for compound (4v):
Figure caption: (4c) The molecular structure of KA181, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
X-ray crystallography study

X-ray data for the compound was collected at room temperature on a Bruker D8 QUEST instrument with an Iμs Mo microsource (λ = 0.7107 Å) and a PHOTON-100 detector. The raw data frames of KA181 were reduced and corrected for absorption effects using the Bruker Apex 3 software suite programs [1]. The structure was solved using intrinsic phasing method [2] and further refined with the SHELXL [2] program and expanded using Fourier techniques. Anisotropic displacement parameters were included for all non-hydrogen atoms. All C bound H atoms of were positioned geometrically and treated as riding on their parent C atoms [C-H = 0.93-0.97 Å, and Uiso(H) = 1.5Ueq(C) for methyl H or 1.2Ueq(C) for other H atoms].

Crystal Data for KA181: C23H18FNO4 (M = 391.38 g/mol): triclinic, space group P-1 (no. 2), a = 8.66220(10) Å, b = 10.7712(2) Å, c = 11.1760(2) Å, α = 102.4181(5)°, β = 97.3598(5)°, γ = 113.4266(4)°, V = 907.47(3) Å³, Z = 2, T = 294.15 K, μ(MoKα) = 0.105 mm⁻¹, Dcalc = 1.432 g/cm³, 31589 reflections measured (4.31° ≤ 2Θ ≤ 56.928°), 4553 unique (Rint = 0.0238, Rsigma = 0.0189) which were used in all calculations. The final R1 was 0.0457 (I > 2σ(I)) and wR2 was 0.1321 (all data). CCDC 1584703 contains supplementary Crystallographic data for the structure. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0) 1223 336 033; email: deposit@ccdc.cam.ac.uk].


Figure caption: The molecular structure of KA181, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.