Iodine promoted dual oxidative C (sp³) - H amination of 2-methyl-3-aryl quinazolin-4(3H)-ones: A facile route to 1,4-diarylimidazo [1,5-a]quinazoline-5(4H)-ones

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X-ray crystallographic information and data

Figure caption: The molecular structure of KA129 with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.

Crystal data for KA129: C_{23}H_{13}ClN_{3}O, M = 439.81, crystal size 0.43 x 0.32 x 0.22 mm^3, triclinic, space group P\overline{1} (No. 2), a = 9.584(5), b = 9.866(4), c = 11.366(6)Å, α = 80.384(9), β = 84.059(12), γ = 66.028(10)°, V = 967.5(8) Å^3, Z = 2, De = 1.510 g/cm^3, F_{000} = 448, PHOTON 100 area detector, MoKa radiation, λ = 0.71073 Å, T = 293(2)K, 2θ_{max} = 61°, 24696 reflections collected, 5909 unique (R_{int} = 0.019), Final Goof = 1.05, RI = 0.0424, wR2 = 0.1245, R indices based on 4261 reflections with I >2σ(I) (refinement on F^2), 280 parameters, μ = 0.247 mm^{-1}, Min. and Max. Resd.Dens. = -0.35, 0.71 e/Å^3. CCDC 1579886 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://www.ccdc.cam.ac.uk/structures/

Data collection and structure solution of KA129: Single crystal X-ray data were collected at room temperature on a Bruker D8 QUEST equipped with a four circle kappa diffractometer and Photon 100 detector. An 1μm microfocus Mo source (λ=0.71073Å) supplied the multi-mirror monochromated incident beam. A combination of Phi and Omega scans were used to collect the necessary data. Unit cell dimensions were determined using 9954 reflections. Integration and scaling of intensity data were accomplished using SAINT program. The structures were solved by Direct Methods using SHELXS97 and refinement was carried out by full-matrix least-squares technique using SHELXL-2014/7. Anisotropic displacement parameters were included for all non-hydrogen atoms. All H atoms were positioned geometrically and treated as riding on their parent C atoms with C-H distances of 0.93--0.97 Å, and with U_{iso}(H) = 1.2U_{eq} (C) or 1.5U_{eq} for methyl atoms.


Copies of spectra

$^1$HNMR (400 MHz, CDCl$_3$) spectrum of compound 3a

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3a
$^1$HNMR (400 MHz, CDCl$_3$) spectrum of compound 3b

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3b
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3c

$^{13}$C NMR (125 MHz, CDCl$_3$) spectrum of compound 3c
$^{1}$HNMR (500 MHz, CDCl$_{3}$) spectrum of compound 3d

$^{13}$C NMR (125 MHz, CDCl$_{3}$) spectrum of compound 3d
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3e

$^{13}$C NMR (125 MHz, CDCl$_3$) spectrum of compound 3e
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3f

$^{13}$C NMR (125 MHz, CDCl$_3$) spectrum of compound 3f
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3g

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3g
$^{1}$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3h

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3h
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3i

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3i
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3j

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3j
$^{1}$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3k

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3k
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3l

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3l
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3m

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3m
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3n

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3n
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3o

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3o
$\text{\textsuperscript{1}H NMR (500 MHz, CDCl$_3$) spectrum of compound 3p}$

$\text{\textsuperscript{13}C NMR (100 MHz, CDCl$_3$) spectrum of compound 3p}$
$\text{H NMR (500 MHz, CDCl}_3\text{) spectrum of compound 3q}$

$\text{C NMR (100 MHz, CDCl}_3\text{) spectrum of compound 3q}$
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3r

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3r
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3s

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3s
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3t

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3t
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3u

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3u
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3v

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3v
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3w

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3w
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3x

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3x
$^{1}H$ NMR (500 MHz, CDCl$_3$) spectrum of compound 3y

$^{13}C$ NMR (100 MHz, CDCl$_3$) spectrum of compound 3y
$^1$H NMR (500 MHz, CDCl$_3$) spectrum of compound 3z

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of compound 3z