Electronic Supplementary Information

Ag-mediated cascade decarboxylative coupling and annulation: A convenient route to 2-Phosphinobenzof[b]phosphole Oxides

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Crystallographic detail of 3m:
1-(4-(2-(diphenylphosphoryl)-1-oxido-1-phenyl-1H-phosphindol-3-yl)phenyl)ethanone

Single crystal X-ray structure of compound 3m, showing 50% probability displacement ellipsoids (arbitrary spheres for H atoms). The compound 3m crystallizes with two molecules in the asymmetric unit. Compound 3m crystallizes in the triclinic P -1 space group with two molecules per unit cell. There is one independent molecule in the asymmetric unit of 3m.

Detector with graphite-monochromated MoKa radiation (λ = 0.71073 Å) at 173 K. All of the Data were corrected for absorption effects using the multi-scan technique. The structures were solved by direct methods, expanded by difference Fourier syntheses and refined by full matrix least-squares on F2 using Bruker SHELXTL (Version 6.10) program package. Non-H atoms were refined anisotropically unless otherwise stated. Hydrogen atoms were introduced at their geometric positions and refined as riding atoms unless otherwise stated. SQUEEZE removed two disordered methanol molecules per formula unit. Some crystal data for 3m: C_{34}H_{26}O_{3}P_{2}, M = 544.49, triclinic, space group P -1, pale yellow plate, a = 10.554(2) Å, b = 12.782(3) Å, c = 12.959(3) Å, α = 75.259(4)°, β = 67.900(4)°, γ = 76.462(4)°, V = 1565.5(6) Å³, Z = 2, Dc = 1.155 g/cm³, F(000) = 568, μ(Mo-Kα) = 0.142 mm⁻¹, T = 173(2) K. Final R [4812 with I > 2σ(I)] = 0.0519, wR2 (all data) = 0.1387. Further details on the crystal structure investigation have been deposited at the Cambridge Crystallographic Data Centre as the deposition number CCDC 1046595.
NMR Spectrum of 3a-3v, 5 and 6:

$^1$H NMR of 3a

$^{13}$C NMR of 3a
$^{31}$P NMR of 3a

$^1$H NMR of 3b
$^{13}$C NMR of 3b

$^{31}$P NMR of 3b
$^1$H NMR of 3c

$^{13}$C NMR of 3c
$^{31}$P NMR of 3c

![31P NMR spectrum of 3c](image)

$^1$H NMR of 3d

![$^1$H NMR spectrum of 3d](image)
$^{13}$C NMR of 3d

$^{31}$P NMR of 3d
$^1$H NMR of 3e

$^{13}$C NMR of 3e
$^{31}$P NMR of 3e

$^1$H NMR of 3f
$^{13}$C NMR of 3f

$^{31}$P NMR of 3f
$^1$H NMR of 3g

$^{13}$C NMR of 3g
$^{31}$P NMR of 3g

$^1$H NMR of 3h
$^{13}$C NMR of 3h

$^{31}$P NMR of 3h
$^1$H NMR of 3i

$^{13}$C NMR of 3i
$^{31}$P NMR of 3i

$^1$H NMR of 3j
$^{13}$C NMR of 3j

$^{31}$P NMR of 3j
$^1$H NMR of 3k

$^{13}$C NMR of 3k
$^{31}$P NMR of 3k

$^1$H NMR of 3l
$^{13}$C NMR of 3l

$^{31}$P NMR of 3l
$^1$H NMR of 3m

$^{13}$C NMR of 3m
$^{31}$P NMR of 3m

$^1$H NMR of 3n
$^{13}$C NMR of 3n

$^{31}$P NMR of 3n
$^1$H NMR of 3o

$^{13}$C NMR of 3o
$^{31}$P NMR of 3o

$^1$H NMR of 3p
$^{13}$C NMR of 3p

$^{31}$P NMR of 3p
$^1$H NMR of 3q

$^{13}$C NMR of 3q
$^{31}$P NMR of $3q$

$^1$H NMR of $3r$
$^{13}$C NMR of 3r

$^{31}$P NMR of 3r
$^1$H NMR of 3s

$^{13}$C NMR of 3s
31 P NMR of 3s

$\text{Ph}_2\text{P(Ph)_2O}$

1 H NMR of 3t
$^1$H NMR of 3u

$^{13}$C NMR of 3u
$^{31}$P NMR of 3u

$^1$H NMR of 3v
\[13^C\text{ NMR of 3v}\]

\[31^P\text{ NMR of 3v}\]
$^1$H NMR of 5

$^{13}$C NMR of 5
$^{31}$P NMR of 5

![Phosphorus NMR Spectrogram of 5]

$^1$H NMR of 6

![Hydrogen NMR Spectrogram of 6]
$^{13}$C NMR of 6

31P NMR of 6