Supplementary Information

One-pot Synthesis of 2-Naphthols from Nitrones and MBH Adducts via Decarboxylative N–O Bond Cleavage

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General methods

Commercially available reagents were used without additional purification, unless otherwise stated. Nitrone **1a** was purchased from Sigma-Aldrich. Nitrones **1b–1v** and Morita-Baylis-Hillman adducts (MBH adducts) **2a** and **2b** were prepared according to the reported literatures.^[1,2] Sealed tubes $(13 \times 100 \text{ mm}^2)$ were purchased from Fischer Scientific and dried in oven for overnight and cooled at room temperature prior to use. Thin layer chromatography was carried out using plates coated with Kieselgel 60 F₂₅₄ (Merck). For flash column chromatography, E. Merck Kieselgel 60 (230–400 mesh) was used. Nuclear magnetic resonance spectra (¹H and ¹³C NMR) were recorded on a Bruker Unity 400 and 500 spectrometers in CDCl₃ and CD₃OD solution and chemical shifts are reported as parts per million (ppm). Resonance patterns are reported with the notations s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), dt (doublet of triplets), br s (broad singlet) and m (multiplet). Coupling constants (*J*) are reported in hertz (Hz). IR spectra were recorded on a Varian 2000 Infrared spectrophotometer and are reported as cm⁻¹. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-600 spectrometer.

Density Functional Theory (DFT) calculation data

All the calculations were performed with Gaussian 09 software.¹ Geometry optimization of all species was carried out with the B3LYP functional² using the 6-31G(d) basis set. Frequency analysis was conducted to ensure the stationary point as minimum or transition state. For the solvent effect of dichloroethane, the single-point calculation on the optimized geometry with SMD solvation model.³ The single-point calculation was carried out by using B3LYP-D3⁴ with a 6-311+G(d,p) basis set.

- Cartesian coordinates of optimized structures (Å)

6**d**

Lowest three frequencies(cm⁻¹): 43.4106, 48.7947, 59.4029

C	1 30288/00	0.015/2200	-0 23731300
C C	3 21574000	0.63240600	0.66446800
C	1 073/8000	0.03249000	0.42784400
C	1.97548000	1 1081/200	0.24205000
C	2.02706200	-1.19814200	0.24393900
C	1 32680200	1 20881000	0.00812400
С и	4.32080200	-1.20881000	0.43248300
п	3.33402700	0.48028300	-0.42073300
П	3.03999000	-2.70313400	1.18800100
C	0.07084800	0.69290600	-0.85062700
H	0.84857300	1.49119200	-1.5/809800
N	0.0162/200	1.24461400	0.35935100
C	-0.85472800	2.45456000	0.21852800
C	0.09784000	3.62910600	-0.07379000
Н	-0.46111100	4.57080800	-0.03966300
Н	0.89828800	3.67592100	0.67263800
Н	0.55752200	3.55719600	-1.06560900
С	-1.51079500	2.68409800	1.59203700
Н	-2.05103600	3.63747900	1.59256600
Н	-2.22015900	1.88734300	1.83102700
Н	-0.75158600	2.71650100	2.38151300
0	-0.81081600	0.11826500	0.85251500
С	-1.94378000	2.37023500	-0.86596300
Н	-2.60238900	1.51426000	-0.69376100
Н	-2.55781500	3.27866000	-0.84599700
Н	-1.51595400	2.29057800	-1.87192300
С	0.55846300	-1.86898900	0.49002400
Н	0.53059100	-2.84389700	-0.00907200
Н	0.41768100	-2.06209100	1.56071800
С	-0.62369700	-1.01619300	-0.00819300
C	-0.28678700	-0.39332000	-1.37077400
H	0.19580400	-1.09979800	-2.05022900
	0117200100	1.0/////000	

Н	-1.17323700	0.02397500	-1.84926600
С	-1.91978000	-1.83085300	-0.04339200
0	-1.95163300	-3.04386000	-0.11826100
0	-3.01409500	-1.05723000	-0.02966100
С	-4.27511900	-1.75329000	-0.11700100
Н	-4.40036300	-2.42454100	0.73656300
Н	-5.03747700	-0.97394000	-0.10185100
Н	-4.33491800	-2.32695700	-1.04577700
Н	3.25909600	1.58607100	-1.18647700
Н	5.23722500	-1.69711300	0.77051300

Int1

Lowest	three frequencies(cm ⁻¹): 41.3531	, 55.8873, 73.2557
С	-3.90986100	-1.29484700	-0.22392600
С	-2.60299600	-1.47443000	-0.67699300
С	-1.63963100	-0.48377200	-0.45671600
С	-1.96979500	0.70266100	0.22932400
С	-3.28541800	0.86389000	0.68027200
С	-4.24743600	-0.12300200	0.45869700
Н	-4.65796300	-2.06244000	-0.40082100
Н	-3.55677300	1.77425200	1.20991300
С	-0.21259500	-0.65359900	-0.92905600
Η	-0.08585900	-1.49119300	-1.61226800
Ν	0.65097100	-0.92400700	0.32038400
С	1.88940500	-1.84713600	0.23797000
С	1.36549800	-3.24821400	-0.09821000
Η	2.20342100	-3.95016200	-0.04475700
Η	0.60606800	-3.57981100	0.62009100
Η	0.94763800	-3.30857800	-1.10765900
С	2.51467100	-1.82993800	1.63954900
Η	3.34419600	-2.54307100	1.65782100
Η	2.90279400	-0.84112300	1.89301700
Η	1.79137800	-2.13501900	2.40572900
0	1.02933100	0.37401300	0.81425000
С	2.86701700	-1.32014900	-0.80988900
Η	3.11322800	-0.27024400	-0.62330500
Η	3.78485500	-1.91373000	-0.74936000
Η	2.47177800	-1.42478500	-1.82562200
С	-0.92629300	1.78814100	0.45204400
Η	-1.22990100	2.71266100	-0.04973200
Η	-0.85251800	2.03642400	1.51747200
С	0.46201100	1.41295400	-0.07085800
С	0.35743700	0.67996500	-1.40489000
Η	-0.32978200	1.17010300	-2.09853700
Η	1.33674600	0.58224200	-1.87219300
С	1.51732600	2.59366400	-0.09355500
0	1.04215100	3.73453000	0.09806600
0	2.69416000	2.24105500	-0.33938500
Η	-2.32852800	-2.38088700	-1.21133500

Η	-5.26289000	0.02388900	0.81713100
Н	0.04414500	-1.32438900	1.04571700

Int1-TS

Lowest three frequencies(cm⁻¹): -433.2132, 53.1145, 63.9309

С	-3.91734200	-1.18480100	-0.30413700
С	-2.60397200	-1.39485200	-0.72344000
С	-1.61481700	-0.44140900	-0.45512300
С	-1.93092800	0.73915900	0.24476300
С	-3.25470800	0.93359100	0.66143300
С	-4.23986200	-0.01749400	0.39409500
Н	-4.68295800	-1.92506400	-0.51945700
Н	-3.51287700	1.84276300	1.20003200
С	-0.17393500	-0.65697000	-0.87419200
Н	-0.07096000	-1.41368200	-1.65513500
Ν	0.60434300	-1.07429000	0.30869000
С	1.74998100	-2.04079600	0.25727500
С	1.17110200	-3.43634600	-0.04746800
Н	1.97963700	-4.17460200	-0.01035500
Н	0.41459100	-3.72353100	0.69186400
Н	0.72033600	-3.47912300	-1.04417900
С	2.38820100	-2.03174100	1.65481200
Н	3.19218400	-2.77336800	1.68909400
Н	2.80779800	-1.05068400	1.89025900
Н	1.65426700	-2.29354700	2.42686800
0	1.20171000	0.57714200	0.81878000
С	2.76730900	-1.62237300	-0.80718500
Н	3.14944400	-0.61736900	-0.61262800
Н	3.60700800	-2.32430700	-0.78235700
Н	2.34225300	-1.65430000	-1.81565700
С	-0.86931500	1.80118100	0.51087300
Н	-1.16871700	2.73665100	0.02582800
Н	-0.80499000	2.01929100	1.58320400
С	0.51973100	1.39676400	0.00263200
С	0.44330500	0.70407900	-1.34187500
Н	-0.21280300	1.18780100	-2.07022900
Н	1.43574200	0.56744300	-1.76849900
С	1.63990400	2.78995000	-0.13399600
0	2.68997600	2.47954300	-0.67972000
0	1.11676300	3.79315000	0.33745400
Н	-2.34222200	-2.29962000	-1.26735300
Н	-5.26024900	0.15361400	0.72713500
Н	0.00659400	-1.28467200	1.10782100

Int2

Lowest three frequencies(cm⁻¹): 15.0726, 24.3776, 32.5054

С	1.77619800	-3.68694400	0.11975500
С	2.00176700	-2.38230900	-0.32178300
С	0.97201200	-1.43411100	-0.31195500
С	-0.30589200	-1.80097700	0.14843700
С	-0.52669500	-3.11475500	0.57957400
С	0.50600800	-4.05379300	0.57093300
Н	2.58373000	-4.41423900	0.10397700
Н	-1.51738100	-3.40303100	0.92452100
С	1.20290000	-0.00866200	-0.78657900
Н	2.09017000	-0.00026800	-1.43327300
Ν	1.37314700	0.88279400	0.38924200
С	2.38942500	1.96986000	0.32690300
С	3.82390300	1.42635800	0.15120700
Н	4.55719300	2.24220300	0.16093900
Н	4.07883000	0.73568300	0.96506000
Н	3.94431600	0.89131100	-0.79793500
С	2.29060800	2.70514400	1.67438800
Н	3.01219300	3.52859500	1.72473700
Н	1.28489600	3.11518700	1.81966900
Н	2.50255000	2.02427500	2.50924100
0	-2.20279800	1.16462900	-0.96547500
С	2.06130000	2.94943000	-0.81028700
Н	1.04034900	3.33536300	-0.71297200
Н	2.75187700	3.80029300	-0.78247300
Н	2.16154300	2.48217700	-1.79635500
С	-1.41931200	-0.77630600	0.19018100
Н	-2.39901800	-1.24846700	0.04940700
Н	-1.45831700	-0.29586100	1.17915100
С	-1.29353700	0.36194800	-0.81737100
С	-0.01367800	0.43850200	-1.62159800
Н	-0.12602100	-0.23436700	-2.48590300
Н	0.09610400	1.45367100	-2.00758600
С	-4.62966700	0.99229000	0.42530000
0	-4.23963300	1.70299200	1.26756200
0	-5.07647700	0.27681600	-0.38463600
Н	2.98605900	-2.09422600	-0.68420400
Н	0.31735600	-5.06965800	0.90833600
Н	1.62076300	0.30226100	1.18874700

Int3

Lowest three frequencies(cm⁻¹):

С	2.90836000	-2.13654100	0.33364500
С	1.61247000	-1.76115300	0.69220900
С	1.11459400	-0.49665500	0.35764700
С	1.93054000	0.40790400	-0.34680000
С	3.23238900	0.02703000	-0.69420800
С	3.72093100	-1.23762400	-0.36131800
Н	3.28332400	-3.12063000	0.60253900

Н	3.86833300	0.72904300	-1.22921800
С	-0.29341600	-0.07056700	0.74020100
Н	-0.63637000	-0.71335800	1.56144300
Ν	-1.18395300	-0.17344500	-0.44510900
С	-2.56965700	-0.68521300	-0.25959200
С	-2.59902300	-2.14209400	0.25150800
Н	-3.62909800	-2.51369100	0.31704600
Н	-2.04611400	-2.80285000	-0.42823300
Н	-2.15582500	-2.23545600	1.24965300
С	-3.22094500	-0.62763200	-1.65227600
Н	-4.25409400	-0.99191600	-1.61646600
Н	-3.22809100	0.39928700	-2.03460000
Н	-2.67120200	-1.25194700	-2.36881700
0	-0.05349200	3.48384200	0.09497800
С	-3.35429400	0.21897400	0.70278200
Н	-3.33317700	1.26173000	0.36650600
Н	-4.40098500	-0.10344800	0.74811800
Н	-2.95592300	0.17610300	1.72262900
С	1.38918900	1.76753300	-0.73258600
Н	2.19180400	2.50942300	-0.81474100
Н	0.92259000	1.72048900	-1.72799700
С	0.31490100	2.32544200	0.19875500
С	-0.25022600	1.38534300	1.24515500
Н	0.40408000	1.44386300	2.12889100
Н	-1.23513200	1.74985800	1.54422000
Н	0.97960400	-2.45351600	1.24309100
Н	4.73427400	-1.51696700	-0.63801400
Н	-0.73727100	-0.79112700	-1.12084600

CO_2

Lowest three frequencies(cm⁻¹): 623.9334, 623.9334, 1373.9776

С	0.00000000	0.00000000	0.00000000
0	0.00000000	0.00000000	1.16895400
0	0.00000000	0.00000000	-1.16895400

References

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 Yang, W. T.; Parr, R. G. Phys. Rev. B 1988, 37, 785.
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X-ray crystallographic data of compound 3pa (CCDC 1858383)

A colorless block-like specimen of $C_{11}H_9FO_2$, approximate dimensions 0.140 mm x 0.180 mm x 0.200 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.



Empirical formula	C11 H9 F O2	
Formula weight	192.18	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 2 ₁ 2 ₁ 2 ₁	
Unit cell dimensions	a = 4.9693(3) Å	$\alpha = 90^{\circ}$.
	b = 5.8579(3) Å	$\beta = 90^{\circ}$.
	c = 31.3863(17) Å	$\gamma = 90^{\circ}$.
Volume	913.64(9) Å ³	
Z	4	
Density (calculated)	1.397 Mg/m ³	
Absorption coefficient	0.109 mm ⁻¹	
F(000)	400	
Crystal size	0.190 x 0.180 x 0.050 r	mm ³
Theta range for data collection	2.596 to 28.759°.	
Index ranges	-6<=h<=6, -7<=k<=7,	-42<=l<=42
Reflections collected	29989	
Independent reflections	2375 [R(int) = 0.0540]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Multi-scan	
Refinement method	Full-matrix least-squar	es on F ²
Data / restraints / parameters	2375 / 0 / 129	
Goodness-of-fit on F ²	1.064	
Final R indices [I>2sigma(I)]	R1 = 0.0506, wR2 = 0.	0992
R indices (all data)	R1 = 0.0690, wR2 = 0.	1060
Absolute structure parameter	-0.5(3)	
Largest diff. peak and hole	0.142 and -0.145 e.Å ⁻³	

Table S1. Crystal data and structure refinement for 3pa.

	Х	у	Z	U(eq)	
C(1)	2684(6)	4479(4)	1454(1)	48(1)	
C(2)	966(6)	4193(4)	1780(1)	50(1)	
C(3)	-734(5)	2270(4)	1779(1)	45(1)	
C(4)	-682(5)	753(4)	1447(1)	41(1)	
C(5)	1106(5)	1100(4)	1102(1)	36(1)	
C(6)	1219(5)	-435(4)	752(1)	40(1)	
C(7)	3000(5)	-74(4)	427(1)	40(1)	
C(8)	4777(5)	1784(5)	432(1)	49(1)	
C(9)	4706(5)	3297(5)	762(1)	50(1)	
C(10)	2871(5)	3005(4)	1100(1)	41(1)	
C(11)	-4045(7)	136(5)	2155(1)	60(1)	
F(1)	4397(4)	6295(3)	1458(1)	72(1)	
O(1)	-2340(4)	2084(4)	2131(1)	58(1)	
O(2)	3180(4)	-1498(3)	78(1)	53(1)	

Table S2. Atomic coordinates (Å x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for 3pa. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

C(1)-C(2)	1.344(4)
C(1)-F(1)	1.362(3)
C(1)-C(10)	1.409(4)
C(2)-C(3)	1.408(4)
C(2)-H(2)	0.9300
C(3)-O(1)	1.367(3)
C(3)-C(4)	1.371(3)
C(4)-C(5)	1.416(3)
C(4)-H(4)	0.9300
C(5)-C(10)	1.419(3)
C(5)-C(6)	1.419(3)
C(6)-C(7)	1.367(3)
C(6)-H(6)	0.9300
C(7)-O(2)	1.380(3)
C(7)-C(8)	1.401(3)
C(8)-C(9)	1.364(4)
C(8)-H(8)	0.9300
C(9)-C(10)	1.410(4)
C(9)-H(9)	0.9300
C(11)-O(1)	1.423(3)
C(11)-H(11A)	0.9600
C(11)-H(11B)	0.9600
C(11)-H(11C)	0.9600
O(2)-H(2A)	0.8200
C(2)-C(1)-F(1)	119.1(2)
C(2)-C(1)-C(10)	124.4(2)
F(1)-C(1)-C(10)	116.4(2)
C(1)-C(2)-C(3)	118.7(2)
C(1)-C(2)-H(2)	120.7
C(3)-C(2)-H(2)	120.7
O(1)-C(3)-C(4)	125.0(2)
O(1)-C(3)-C(2)	114.4(2)
C(4)-C(3)-C(2)	120.6(2)

Table S3. Bond lengths [Å] and angles [°] for 3pa.

C(3)-C(4)-C(5)	120.1(2)
C(3)-C(4)-H(4)	120.0
C(5)-C(4)-H(4)	120.0
C(4)-C(5)-C(10)	120.2(2)
C(4)-C(5)-C(6)	121.6(2)
C(10)-C(5)-C(6)	118.2(2)
C(7)-C(6)-C(5)	120.2(2)
C(7)-C(6)-H(6)	119.9
C(5)-C(6)-H(6)	119.9
C(6)-C(7)-O(2)	122.7(2)
C(6)-C(7)-C(8)	121.4(2)
O(2)-C(7)-C(8)	115.9(2)
C(9)-C(8)-C(7)	119.7(2)
C(9)-C(8)-H(8)	120.1
C(7)-C(8)-H(8)	120.1
C(8)-C(9)-C(10)	120.6(2)
C(8)-C(9)-H(9)	119.7
C(10)-C(9)-H(9)	119.7
C(1)-C(10)-C(9)	124.1(2)
C(1)-C(10)-C(5)	116.0(2)
C(9)-C(10)-C(5)	119.8(2)
O(1)-C(11)-H(11A)	109.5
O(1)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
O(1)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(3)-O(1)-C(11)	116.97(19)
C(7)-O(2)-H(2A)	109.5

Symmetry transformations used to generate equivalent atoms:

	U^{11}	U ²²	U ³³	U ²³	U ¹³	U^{12}
C(1)	49(2)	33(1)	63(2)	-2(1)	-16(1)	-7(1)
C(2)	55(2)	40(1)	55(2)	-12(1)	-13(2)	4(1)
C(3)	45(1)	47(1)	44(1)	-5(1)	-6(1)	5(1)
C(4)	37(1)	42(1)	45(1)	-5(1)	-3(1)	-4(1)
C(5)	32(1)	36(1)	40(1)	1(1)	-10(1)	1(1)
C(6)	37(1)	41(1)	43(1)	-2(1)	-4(1)	-5(1)
C(7)	33(1)	50(1)	38(1)	0(1)	-5(1)	4(1)
C(8)	37(1)	63(2)	48(2)	10(1)	2(1)	-6(1)
C(9)	42(1)	47(1)	61(2)	10(1)	-6(1)	-10(1)
C(10)	39(1)	35(1)	48(1)	3(1)	-11(1)	-2(1)
C(11)	64(2)	65(2)	50(2)	-2(1)	10(2)	-1(2)
F(1)	80(1)	44(1)	94(1)	-9(1)	-12(1)	-24(1)
O (1)	62(1)	65(1)	48(1)	-17(1)	8(1)	-4(1)
O(2)	42(1)	71(1)	47(1)	-13(1)	4(1)	-2(1)

Table S4. Anisotropic displacement parameters (Å²x 10³) for 3pa. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h² a*²U¹¹ + ... + 2 h k a* b* U¹²]

	Х	у	Z	U(eq)	
H(2)	901	5245	2002	60	
H(4)	-1823	-506	1448	49	
H(6)	75	-1692	744	48	
H(8)	6000	1984	211	59	
H(9)	5879	4534	764	60	
H(11A)	-5239	124	1914	89	
H(11B)	-5078	195	2413	89	
H(11C)	-2968	-1224	2153	89	
H(2A)	1735	-2146	42	80	

Table S5. Hydrogen coordinates (Å x 10⁴) and isotropic displacement parameters (Å²x 10 3) for 3pa.



¹H NMR and ¹³C NMR spectra of all compounds













SpinWorks 4: SH.1658











S21



SpinWorks 4: SH.1660_MeOD























SpinWorks 4: SH.1665





































































SpinWorks 4: SH.1742





SpinWorks 4: HY.126





S42

SpinWorks 4: SH.1726_MeOD



SpinWorks 4: SH.1726_MeOD





SpinWorks 4: SH.1728





SpinWorks 4: AP-383-2B



SpinWorks 4: SH.1679 = SH.1618-d

