

Electronic supplementary information (ESI)

Realization of deep-blue TADF in sterically controlled naphthyridines for vacuum- and solution-processed OLEDs

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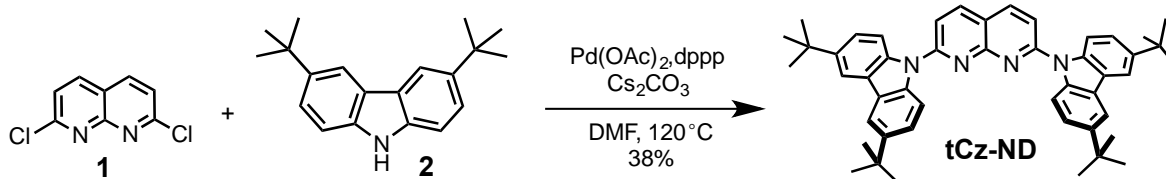
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Synthesis and identification

All reagents and starting materials were obtained from commercial sources and used as received. Reaction solvents, dimethylformamide was distilled from CaH₂, THF was distilled from sodium/ benzophenone. All moisture-sensitive reactions were performed in oven-dried (230 °C) glassware under an atmosphere of dry argon. Thin-layer chromatography was performed on Merck silica gel plates with QF-254 indicator. Visualization was accomplished with UV (254 nm), potassium permanganate (KMnO₄), ninhydrin or vanillin. Column chromatography was performed using Merck silica 60 (40–63 μm particle size). ¹H and ¹³C NMR spectra were recorded on a NMR spectrometer at 400 MHz for ¹H and 101 MHz for ¹³C, respectively. ¹H and ¹³C NMR spectra are referenced to residual solvent (CDCl₃, 7.29 and 77.16 ppm for ¹H NMR and ¹³C NMR, respectively). When necessary, assignments were obtained by reference to COSY, HSQC, and HMBC correlations. Chemical shifts are reported in ppm, and multiplicities are indicated by br (broad), s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sxt (sextet), sept (septet), m (multiplet), and combinations thereof. Infrared (IR) spectra were recorded on a FTIR spectrometer equipped with a diamond ATR unit. Melting points were determined in open capillary tubes and are uncorrected. HRMS was recorded on Bruker Daltonics microTOF-II spectrometer equipped with ESI ion source in positive mode.

2,7-dichloro-1,8-naphthyridine **1**¹ and 1-bromo-3,6-di-*tert*-butyl-9*H*-carbazole **4**² were synthesized according to literature.

Abbreviations: DCM, dichloromethane; DMF, dimethylformamide; dppp, 1,3-bis(diphenylphosphino)propane; EtOAc, ethyl acetate; PE, petrol ether (40–60 °C fraction).



Pd(OAc)₂ (12.2 mg, 5 mol %) and 1,3-bis(diphenylphosphino)propane (dppp) (41.2 mg, 5 mol %) was dissolved in 10 ml DMF at room temperature. After 5 min, 2,7-dichloro-1,8-naphthyridine **1** (100 mg, 1.0 mmol, 1.0 equiv.), 3,6-di-*tert*-butyl-9H-carbazole **2** (765 mg, 3.0 mmol, 3.0 equiv.) and cesium carbonate (1.63 g, 5.0 mmol, 5.0 equiv.) were added. The reaction mixture was heated at 120 °C for 48 h. Reaction was quenched with water. After extraction with CHCl₃, the crude product was purified by column chromatography (EtOAc/PE 1:60) to afford 263 mg (38%) of **tCz-ND** as a light-yellow solid.

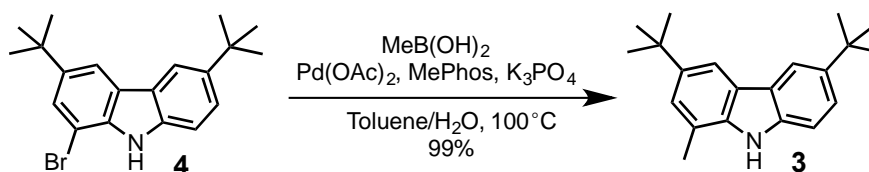
R_f = 0.4 (1/20 EtOAc:PE), m.p. >280°C.

¹H NMR (400 MHz, CDCl₃) δ 8.36 (d, *J*=8.5, 2H), 8.24 (d, *J*=8.7, 4H), 8.15 (d, *J*=1.9, 4H), 7.96 (d, *J*=8.5, 2H), 7.57 (dd, *J*=8.7, 2.0, 4H), 1.51 (s, 36H);

¹³C NMR (100 MHz, CDCl₃) δ 155.9, 154.8, 144.8, 138.6, 137.7, 125.2, 124.22, 118.2, 116.8, 116.1, 112.2, 34.8, 31.9;

IR: ν_{max}/cm⁻¹ 2954, 1470, 1360, 808;

HRMS (ESI) calc. for C₄₈H₅₃N₄ (M+H): 685.4265; Found: 685.4264.



Reaction tube was charged with 1-bromo-3,6-di-*tert*-butyl-9H-carbazole **4** (320 mg, 0.893 mmol, 1.0 equiv.), methylboronic acid (134 mg, 2.239 mmol, 2.5 equiv.), tribasic potassium phosphate (0.76g, 3.58 mmol, 4 equiv.), Pd(OAc)₂ (10 mg, 0.045 mmol, 0.05 equiv.) and MePhos (33 mg, 0.091 mmol, 0.1 equiv.) under argon. Next, degassed solution of toluene/H₂O (3:1, 10 ml) was added. Screw-capped tube was heated at 100°C for 48h. Filtration through celite pad with EtOAc and purification by column chromatography on silica gel DCM/PE (1:5) afforded 259 mg (99%) of **3** as a yellowish oil.

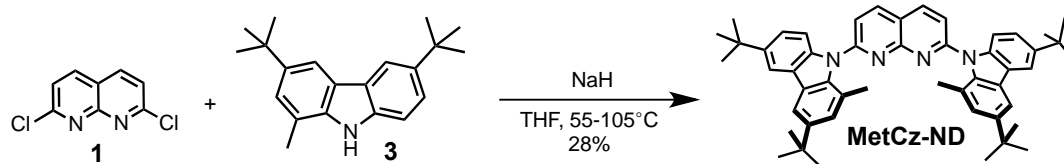
R_f = 0.29 (1/5 DCM:PE).

¹H NMR (400 MHz, CDCl₃) δ 8.08-8.10 (m, 1H), 7.94-7.96 (m, 1H), 7.80 (s, 1H), 7.47-7.50 (m, 1H), 7.38-7.41 (m, 1H), 7.29-7.31 (m, 1H), 2.58 (s, 3H), 1.48 (s, 9H), 1.47 (s, 9H);

^{13}C NMR (100 MHz, CDCl_3) δ 142.5, 142.2, 138.0, 137.5, 124.3, 123.8, 123.4, 122.8, 118.9, 116.3, 113.7, 110.1, 34.7, 34.6, 32.1, 32.0, 17.2;

IR: $\nu_{\text{max}}/\text{cm}^{-1}$ 3421, 2951, 1493, 1296, 865, 805;

HRMS (ESI) calc. for $\text{C}_{21}\text{H}_{28}\text{N}$ (M+H): 294.2216; Found: 294.2208.



To a solution of **3** (161 mg, 0.549 mmol, 2.2 equiv.) in freshly distilled THF (8.0 ml) NaH (60% dispersion in mineral oil) (30 mg, 0.75 mmol, 3.0 equiv.) was added in one portion under argon. Next, mixture was stirred at 55-60°C for 1.5 h and subsequently cooled to r.t. Afterwards **1** (49 mg, 0.249 mmol, 1.0 equiv.) was added in one portion, tube was screw-capped and heated at 105°C for 96h. Purification by column chromatography on silica gel 1/30 (EtOAc:PE) afforded 49 mg (28%) of **MetCz-ND** as a yellowish solid.

R_f = 0.31 (1/20 EA:PE), m.p. >300°C.

^1H NMR (400 MHz, CDCl_3) δ 8.34 (d, J = 8.4 Hz, 2H), 8.10-8.12 (m, 2H), 8.00-8.02 (m, 2H), 7.92 (d, J = 8.4 Hz, 2H), 7.54 (d, J = 8.4 Hz, 2H), 7.46-7.50 (m, 2H), 7.31-7.33 (m, 2H), 2.29 (s, 6H), 1.50 (s, 18H), 1.48 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.6, 155.0, 144.7, 144.6, 140.1, 137.8, 137.7, 127.1, 126.2, 125.0, 124.1, 122.1, 120.7, 119.3, 116.0, 114.0, 111.1, 34.8, 34.6, 31.9, 29.7, 21.3;

IR: $\nu_{\text{max}}/\text{cm}^{-1}$ 2952, 1491, 1446, 1230, 867, 806;

HRMS (ESI) calc. for $\text{C}_{50}\text{H}_{57}\text{N}_4$ (M+H): 713.4578; Found: 713.4577.

X-Ray Crystallography

Single crystals of the investigated compounds were grown from DMF solutions by slow evaporation method. Suitable crystals were selected and mounted on a nylon loop in inert oil and analyzed on a XtaLab Synergy diffractometer equipped with HyPix-6000HE hybrid photon counting detector and PhotonJet microfocus X-ray source delivering $\text{CuK}\alpha$ ($\lambda = 1.54184$) radiation. X-ray measurements were carried out at 100.0 K using Oxford Cryostream 800 cooling system. Data were collected and processed using CrysAlisPro software. Employing Olex2 graphical interface,³ the structures were solved by Intrinsic Phasing with the ShelXT⁴ program and refined with the ShelXL⁵ package using Least Squares minimization. It is worth noting that highly disordered solvent molecules in **MetCz-ND** crystal lattice were present. To remove the electronic contribution of solvent molecules from the refinement, solvent masking was applied. The structure files of **tCz-ND** and **MetCz-ND** crystals were deposited with the Cambridge Crystallographic Data Centre (CCDC 1991494; 1992697).

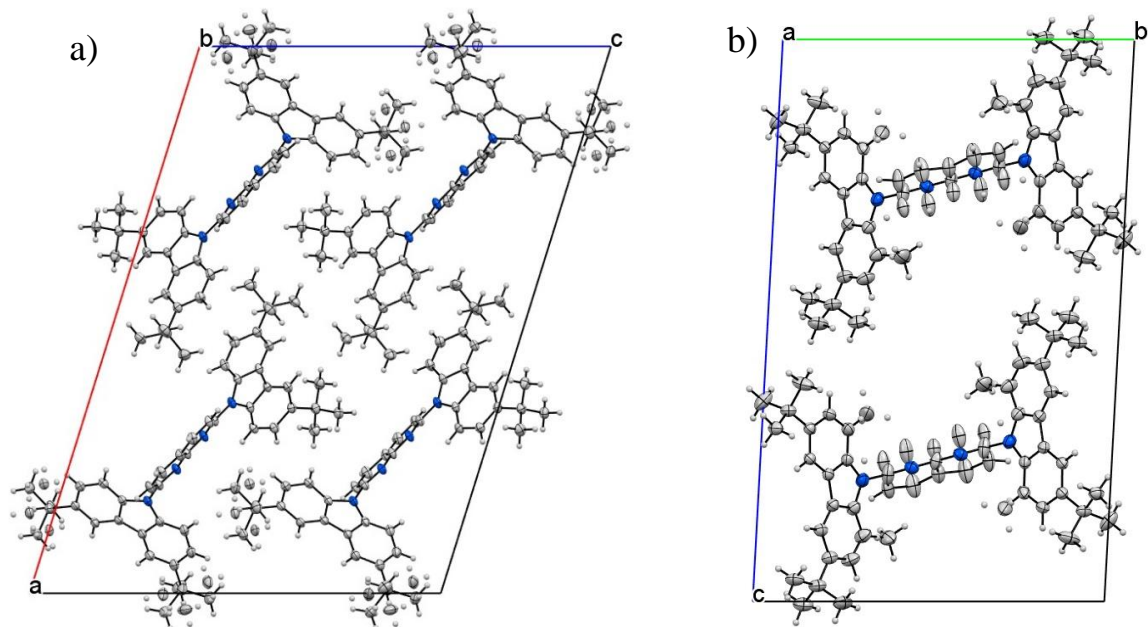


Figure S1. Illustration of unit cell contents obtained by XRD measurements: a) view down crystallographic axis *b* of **tCz-ND**; b) view down crystallographic axis *a* of **MetCz-ND**. Thermal ellipsoids are at 50% probability level.

Table S1. Crystallographic data of **tCz-ND** and **MetCz-ND** crystals.

Compound	tCz-ND	MetCz-ND
Formula	C ₄₈ H ₅₂ N ₄	C ₅₀ H ₅₆ N ₄
$\rho_{calc./g\ cm^{-3}}$	1.181	0.963 + solvent
μ/mm^{-1}	0.524	0.425
Formula Weight	684.93	712.98
Colour	yellow	yellow
Shape	needle	needle
Size/mm ³	0.51×0.05×0.04	0.70×0.06×0.05
T/K	100.00(13)	99.99(11)
Crystal System	monoclinic	triclinic
Space Group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> -1
<i>a</i> /Å	30.1012(6)	5.94900(10)
<i>b</i> /Å	6.18300(10)	16.31420(10)
<i>c</i> /Å	21.6823(4)	25.8129(2)
$\alpha/^\circ$	90	93.0010(10)
$\beta/^\circ$	107.347(2)	90.0120(10)
$\gamma/^\circ$	90	100.5010(10)
<i>V</i> /Å ³	3851.87(13)	2459.77(5)
<i>Z</i>	4	2
<i>Z'</i>	1	1
Wavelength/Å	1.54184	1.54184
Radiation type	Cu K _{α}	Cu K _{α}
$\theta_{min}/^\circ$	3.076	2.759
$\theta_{max}/^\circ$	71.879	76.681
Measured Reflections	21090	44637
Independent Reflections	7334	9455
Reflections with <i>I</i> > 2(<i>I</i>)	6200	7558
<i>R</i> _{int}	0.0245	0.0319
Parameters	544	570
Restraints	0	0
Largest Peak	0.212	0.402
Deepest Hole	-0.202	-0.194
Goodness-of-fit	1.039	1.066
<i>wR</i> ₂ (all data)	0.1115	0.1792
<i>wR</i> ₂	0.1066	0.1709
<i>R</i> ₁ (all data)	0.0497	0.0660
<i>R</i> ₁	0.0410	0.0568

Thermal stability

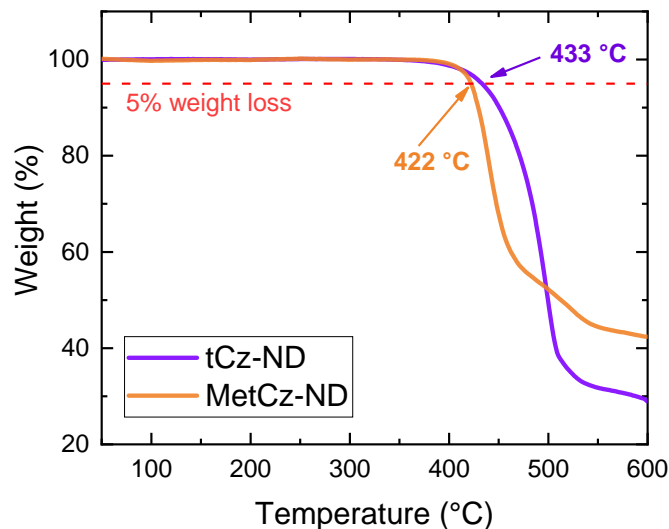


Figure S2. TGA traces of **tCz-ND** and **MetCz-ND** recorded at 10 K/min heating rate in N₂ environment. Thermal decomposition temperatures corresponding to 5% weight loss are indicated.

DFT calculations

Ground state geometries were optimized in vacuum by employing density functional theory (DFT) with B3LYP functional at 6-31G(d) basis set as implemented in Gaussian 09W software.⁶ Time dependent DFT method at the same basis set level was used to calculate ground to excited state transition energies (E), oscillator strengths (f) and to obtain HOMO and LUMO orbitals.

Table S2. Data obtained from TD-DFT calculations at B3LYP/6-31G(d) level by using optimized ground state geometry at the same basis set.

Compound	$E_{S_0 \rightarrow S_1}$ (eV)	f [$S_0 \rightarrow S_1$]	$E_{S_0 \rightarrow S_2}$ (eV)	f [$S_0 \rightarrow S_2$]	$E_{S_0 \rightarrow T_1}$ (eV)	$E_{S_0 \rightarrow T_2}$ (eV)	\angle tCz/ND ^a (deg)	$\Delta E_{S_1-T_1}$ (eV)
tCz-ND	2.9056	0.4963	3.1734	0.0274	2.5559	2.7612	31	0.3497
MetCz-ND	2.7041	0.3100	2.8937	0.0241	2.465	2.6106	55	0.2391

^a dihedral angle between carbazole and naphthyridine

Photophysical properties

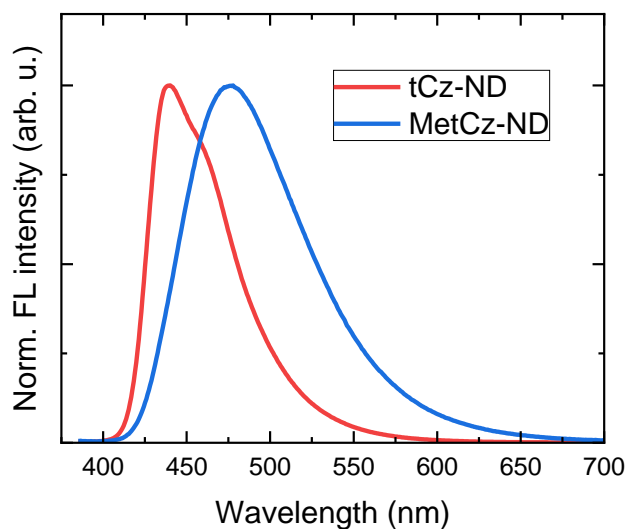


Figure S3. FL spectra of naphthyridine derivatives in toluene solutions (10^{-5} mol/l) at room temperature.

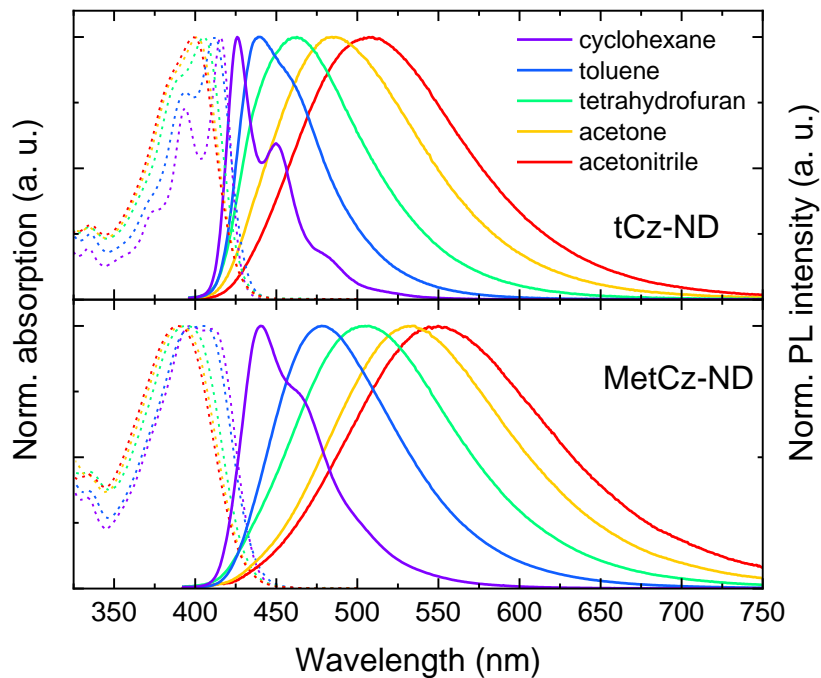


Figure S4. Absorption and FL spectra of naphthyridine derivatives in solvents of different polarity. Solution concentration $c = 10^{-5}$ mol/l.

Table S3. Photophysical properties of naphthyridine compounds in toluene (10^{-5} mol/l) and mCP host (7 wt%)

	Compound	λ_{\max} (nm)	Φ_{FL}	$\Phi_{\text{DF}}/\Phi_{\text{PF}}$	τ_{PF} (ns)	τ_{DF} (μs)	k_r (10^6 s^{-1})	$k_{\text{rISC}}^{\text{a}}$ (10^6 s^{-1})	$k_{\text{ISC}}^{\text{a}}$ (10^7 s^{-1})
Toluene	tCz-ND	440	0.53	0.26	4.0	2.8	104.2	0.16	14.4
	MetCz-ND	477	0.64	0.49	12.1	1.1	35.4	0.81	4.7
7 wt% In mCP	tCz-ND	452	0.76	2.3	5.2	8.8	44.2	0.34	14.8
	MetCz-ND	478	0.86	2.44	8.3	3.1	30.1	1.06	9.0

^a ISC (k_{ISC}) and rISC (k_{rISC}) rates were calculated according to the previously described procedures⁷ assuming that non-radiative decay occurs mainly from the triplet states as determined from the data in Figure 4.

OLED performance

Table S4. Main parameters of vacuum- and solution-processed OLEDs based on the naphthyridine TADF emitters (7 wt% in mCP).

Tech.	Emitter	V_{on} (V)	Maximum values				@100 cd/m^2				@1000 cd/m^2				CIE (x, y)	λ_{max} (nm)	FWHM (nm)
			EQE (%)	L (cd/m^2)	CE (cd/A)	LE (lm/W)	Voltage (V)	EQE (%)	CE (cd/A)	LE (lm/W)	Voltage (V)	EQE (%)	CE (cd/A)	LE (lm/W)			
Vac	tCz-ND	3.25	17.01	8424	22.17	19.88	4.50	9.89	13.41	9.44	6.20	7.00	9.30	4.72	0.14, 0.16	459	66
Sol	tCz-ND	4.3	13.45	6840	17.26	10.24	6.50	12.11	15.84	7.64	8.55	7.80	9.80	3.48	0.15, 0.14	452	66
Vac	MetCz-ND	3.25	17.60	21459	31.98	23.60	4.50	14.43	25.46	17.76	5.87	12.50	24.72	13.2	0.18, 0.32	481	88
Sol	MetCz-ND	3.4	11.66	23028	23.84	11.76	4.87	7.5	14.75	9.80	6.12	11.00	22.80	11.6	0.16, 0.29	479	80

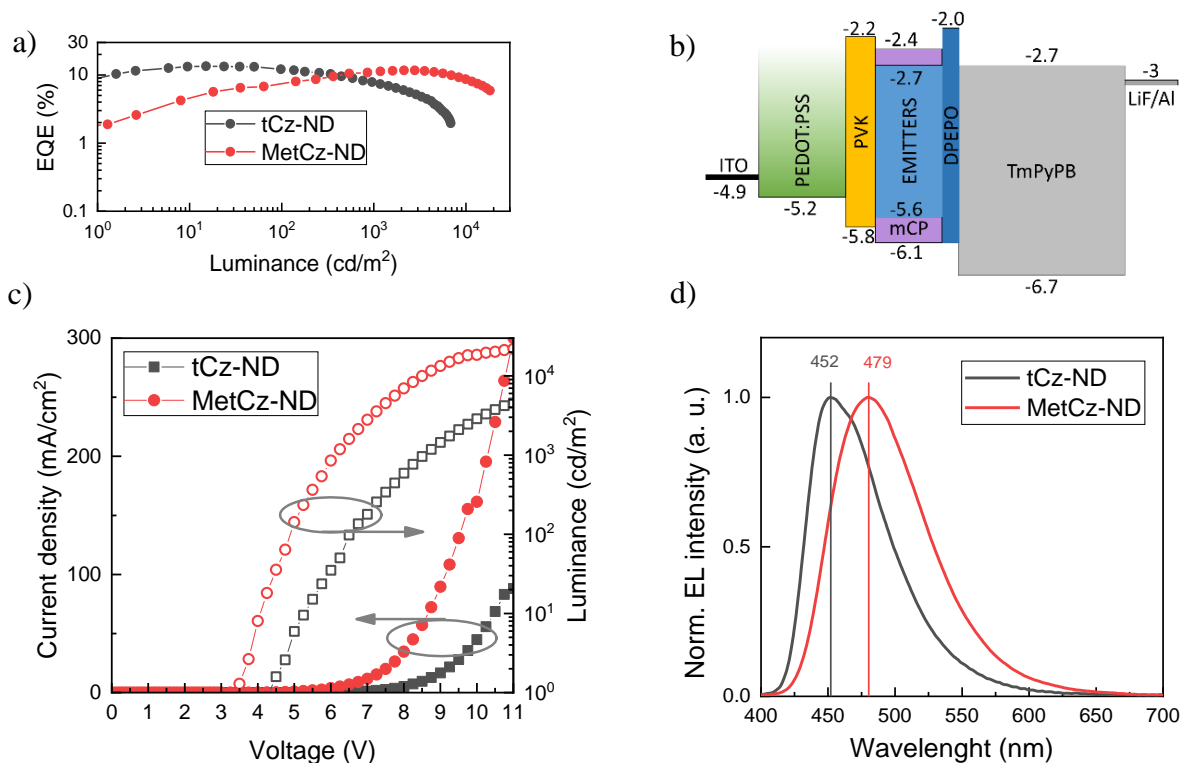


Figure S5. Characteristics of solution-processed OLEDs based on the naphthyridine TADF emitters dispersed in mCP host at 7 wt%: a) EQE vs luminance, b) energy level diagram, c) current density and luminance vs applied voltage, d) normalized electroluminescence spectra.

Table S5. Summarized main EL parameters of the best performing deep-blue ($\lambda_{\text{max}} < 460$ nm) TADF OLEDs reported in literature.

Compound	λ_{max} (nm)	CIE (x; y)	FWHM (nm)	EQE _{max} (%)	EQE (%) @100 cd/m ²	EQE (%) @1000 cd/m ²	Reference
tCz-ND (vacuum processed)	459	(0.14; 0.16)	66	17.0	9.9	7.1	This work
tCz-ND (solution processed)	452	(0.15; 0.14)	66	13.45	12.11	7.8	This work
34CzBN	458	(0.14; 0.14)	63	15.2	7.3	-	8
DCzBN2	436	(0.15; 0.07)	66	7.7	-	-	9
DCzBN3	428	(0.16; 0.06)	65	10.3	-	-	9
CNICCz	449	(0.15; 0.08)	56	12.4	6.4	-	10
CNICtCz	456	(0.14; 0.13)	60	16.0	10.7	-	10
DTTSAF	448	(0.15; 0.10)	68	6.2	~4.6	-	11
BDTPDDA	456	(0.14; 0.12)	56	8.5	~6.6	4.6	11
ICzAc	454	(0.15; 0.09)	56	13.7	-8	-	12
<i>i</i>-2CzdOXDMe	452	(0.17; 0.17)	85	11.8	1.3	-	13
DPAc- DdtCzBN	456	(0.16; 0.15)	67	23.1	18.3	-	14
TXAZ	456	(0.15; 0.13)	69	16.0	8.48	-	15
pDTCz-3DPyS	452	(0.15; 0.13)	~75	13.4	4.5	-	16
CzMeoB	455	(0.14; 0.10)	~50	16.1	12.4	-	17
TDBA-Ac	448	(0.15; 0.06)	48	21.5	~18	9.6	18
DMAC2PTO	448	(0.15; 0.11)	52	15.2	~5	-	19
PhICzACD	454	(0.16; 0.12)	51	17.0	6.4	-	20
DtBuAc-DBT	455	(0.13; 0.13)	84	10.5	9.8	-	21
OBA-O	446	(0.17; 0.17)	~100	17.8	15.5	8.5	22
Cz-TRZ4	446	(0.15; 0.10)	~70	18.3	-	~9	23
tCbz-mPYRs	441	(0.16; 0.12)	80	8.7	2.0	-	24
DCzTrz	459	(0.15, 0.15)	~60	17.5	~16	~11	25
DMTDAc	451	(0.15; 0.13)	65	19.8	19.8	~12	26
DMOC-DPS	460	(0.16; 0.16)	~80	14.5	9.0	3.7	27
Ac-3MHPM	451	(0.16; 0.15)	~75	17.8	10.4	-	28
FA-TA	454	(0.15; 0.13)	~70	11.2	-	-	29
3b	436	(0.16; 0.08)	~60	8.5	8.4	7.7	30
2CzdOXDPh	455	(0.16; 0.15)	75	6.8	2	1.4	31
TN4T-PCZ	~410	(0.16; 0.03)	~70	20.4	~12	20	32

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