Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Visual Organophosphate Vapor Sensing by Dibenzylidine Derivatives Exhibiting Intramolecular Charge Transfer and Aggregation Induced Emission

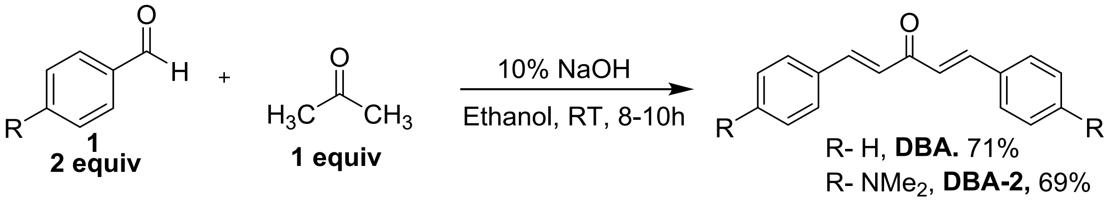
Contents

1. General methods. Flash chromatography was performed using silica gel (230−400 mesh) as the stationary phase. NMR spectra were recorded on Bruker DPX 400 instruments. The Chemical shifts, given in ppm, are relative to the residual solvent peaks. Ultraviolet–visible (UV–vis) spectra were recorded on an Evolution 220 UV–visible spectrometer (Thermo Scientific, Madison, WI). Fluorescence spectroscopy measurements were carried out using a Fluorolog spectrophotometer (HORIBA Scientific, Irvine, CA). Single crystal XRD measurements were carried out using APEX II Bruker diffractometer at temperature 200(2) K. Scanning electron microscopy (SEM) images were recorded on a JEOL JSM-7400F scanning electron microscope (JEOL Ltd, Tokyo, Japan) operated and analyzed using the instrument software. Transmission electron microscopy (TEM) images of the aggregates were obtained on a FEI Tecnai 12 TWIN TEM operating at 120 kV.

2. Synthesis.

2.1 General Procedure A for the synthesis of symmetrical dibenzylidineacetone (DBA) derivatives

This reaction was carried out according to a literature method as described below.[1]



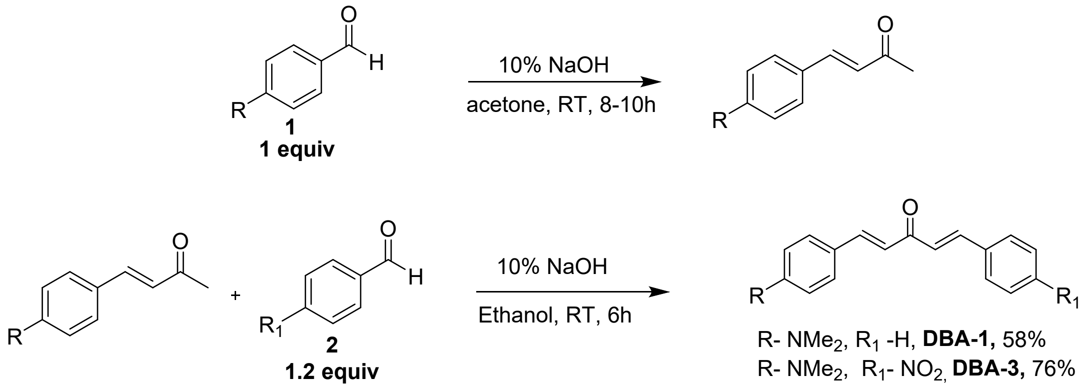
Synthesis of symmetrical **DBA** derivatives.

A solution of ethanol (5 ml) and 10% sodium hydroxide (5 ml) were taken in a round bottomed flask and the substituted benzaldehyde **1** (5 mmol), acetone (2.5 mmol) was added in to the solution then the reaction mixture was stirred at room temperature for 8-10 hours. After the reaction, aq. HCl solution was added to quench the reaction and the aqueous layer was extracted with EA (50 mL × 3). The combined organic layers were washed with brine (50 mL ×1), dried over anhydrous Na_2SO_4 and concentrated. The solvent was removed under reduced pressure and the residue was chromatographed on silica gel (PE/EA) to get the desired product. All the products were confirmed by comparing their ^{1}H NMR data with those reported in previous literature.[1]

Reference:

1.M. Cui, M. Ono, H. Kimura, B. Liu, and H. Saji, *J. Med. Chem.*, 2011, **54**, 2225–2240.

2.2 General Procedure B for the synthesis of unsymmetrical dibenzylidine acetone (DBA) derivatives



Synthesis of unsymmetrical **DBA** derivatives.

A solution of acetone (5 ml) and 10% sodium hydroxide (5 ml) were taken in a round bottomed flask and the substituted benzaldehyde **1** (5 mmol) was added in to the solution then the reaction mixture was stirred at room temperature for 8 hours. After that acetone was evaporated from the solution and the resultant solution was re dissolved in ethanol then the substituted benzaldehyde **2 (**6 mmol) was added in to the same solution and the mixture was further stirred at room temperature for 6 hours. Aq. HCl solution was added to quench the reaction and the aqueous layer was extracted with EA (50 mL × 3). The combined organic layers were washed with brine (50 mL ×1), dried over anhydrous Na_2SO_4 and concentrated. The solvent was removed under reduced pressure and the residue chromatographed on silica gel (PE/EA) to get the desired product. All the products were confirmed by comparing their ^{1}H NMR data with those reported in previous literature.**[1]**

2. 3 Spectral data for the compounds DBA - DBA-3.

DBA: Following the general procedure **A**, the product DBA was obtained as a pale yellow solid and the structure of the compound was confirmed by ^{1}H and ^{13}CNMR data with those reported in previous literature.[1]

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.75 (d, *J* = 15.6 Hz, 2H), 7.63 (dd, *J* = 7.6, 3.8 Hz, 4H), 7.43 – 7.41 (m, 6H), 7.09 (d, *J* = 15.6 Hz, 2H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 189.3, 143.5, 134.9, 130.6, 129.1, 128.5, 125.5.

DBA-1: Following the general procedure **B**, the product DBA-1 was obtained as an orange solid and the structure of the compound was confirmed by ^{1}H and ^{13}CNMR data with those reported in previous literature.[1]

^{1}H NMR (400 MHz, CDCl$_3$) : δ 7.72 (d, *J* = 15.6 Hz, 1H), 7.71 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 7.8 Hz, 2H), 7.52 (d, *J* = 15.6 Hz, 1H), 7.43 – 7.38 (m, 3H), 7.09 (d, *J* = 15.6 Hz, 1H), 6.88 (d, *J* = 15.6 Hz, 1H), 6.70 (d, *J* = 7.6 Hz, 2H), 3.05 (s, 6H).

^{13}C NMR (100 MHz, CDCl$_3$): 190.6, 144.1, 142.4, 132.1, 130.5, 130.3, 129.0, 128.4, 126.0, 111.2, 40.1.

DBA-2: Following the general procedure **A**, the product DBA-2 was obtained as an orange solid and the structure of the compound was confirmed by ^{1}H and ^{13}CNMR data with those reported in previous literature.[1]

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.68 (d, *J* = 15.6 Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 4H), 6.89 (d, *J* = 15.6 Hz, 2H), 6.69 (d, *J* = 7.8 Hz, 4H), 3.03 (s, 12H).

^{13}C NMR (100 MHz, CDCl$_3$): 189.1, 151.93, 143.3, 130.8, 130.4, 123.1, 112.0, 40.3.

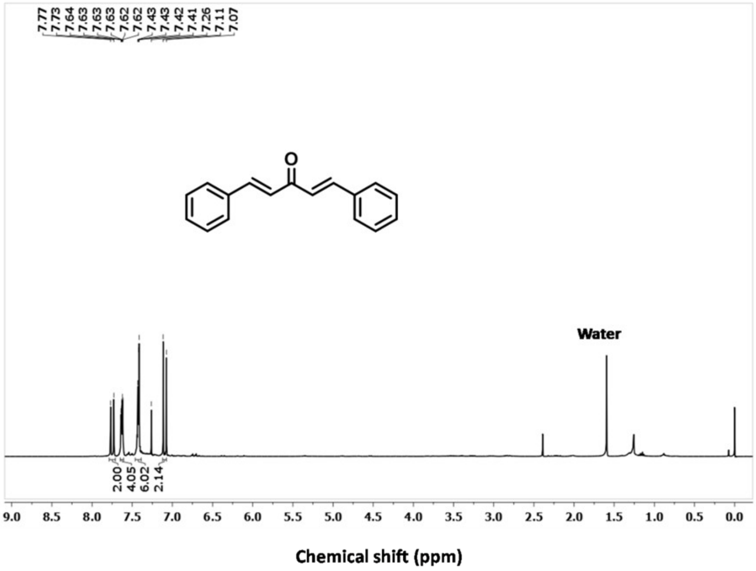
DBA-3: Following the general procedure **B**, the product DBA-3 was obtained as an orange solid and the structure of the compound was confirmed by ^{1}H and ^{13}CNMR data with those reported in previous literature.[1]

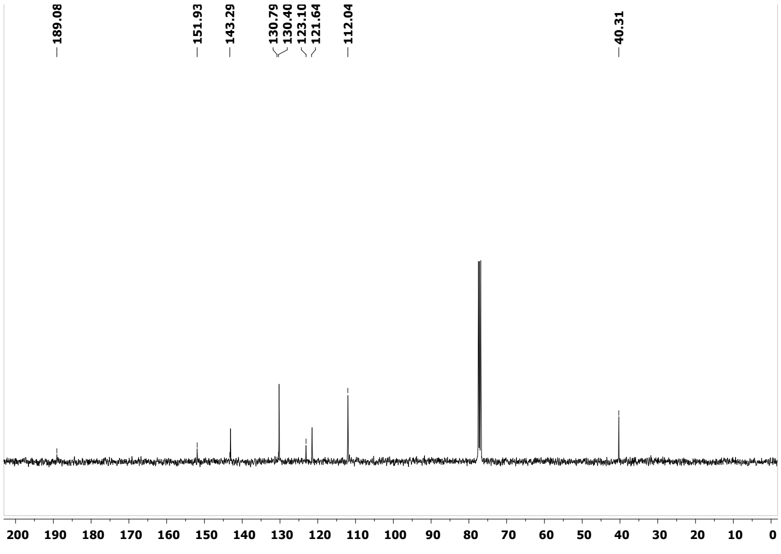
^{1}H NMR (400 MHz, CDCl$_3$) : δ 8.26 (d, *J* = 7.6 Hz, 2H), 7.75 (d, *J* = 15.6 Hz, 1H), 7.74 (d, *J* = 7.6 Hz, 2H), 7.71 (d, *J* = 15.6 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 15.6 Hz, 1H), 6.86 (d, *J* = 15.6 Hz, 1H), 6.70 (d, *J* = 7.6 Hz, 2H), 3.06 (s, 6H).

^{13}C NMR (100 MHz, CDCl$_3$): δ 188.0, 145.4, 139.0, 130.7, 129.5, 128.9, 124.3, 121.3, 112.4, 40.7.

3. NMR data.

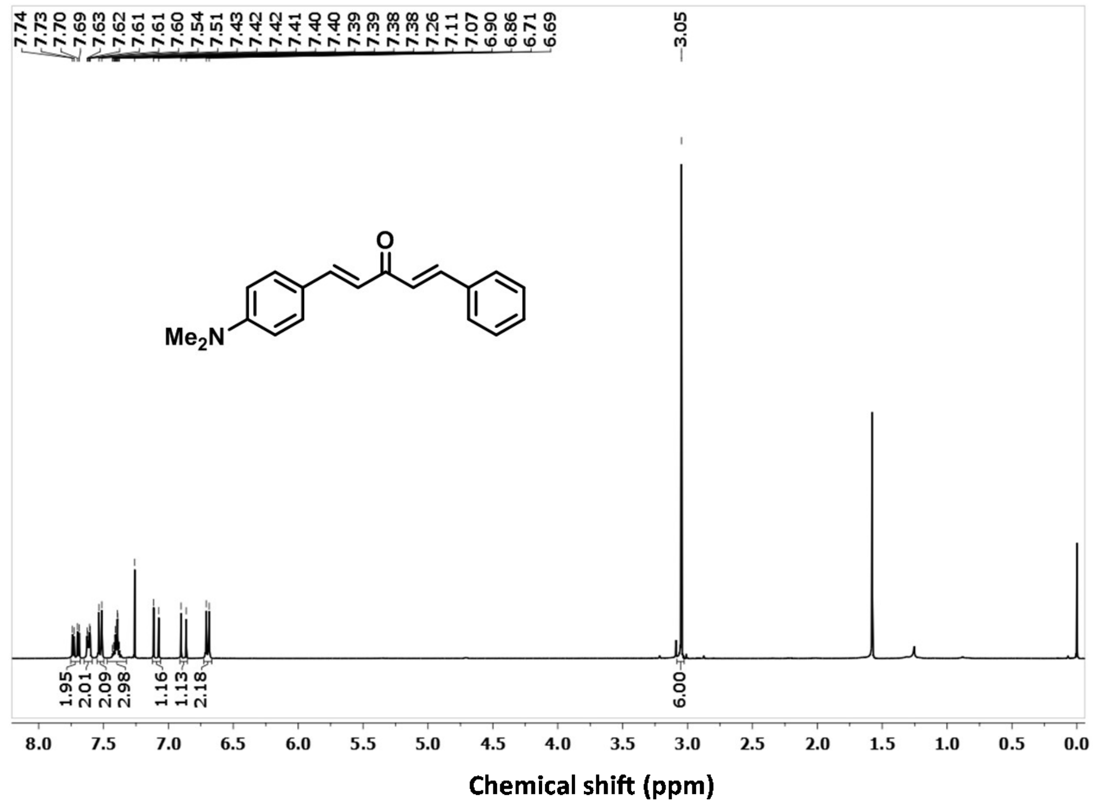
^{1}H and ^{13}CNMR Spectra of DBA

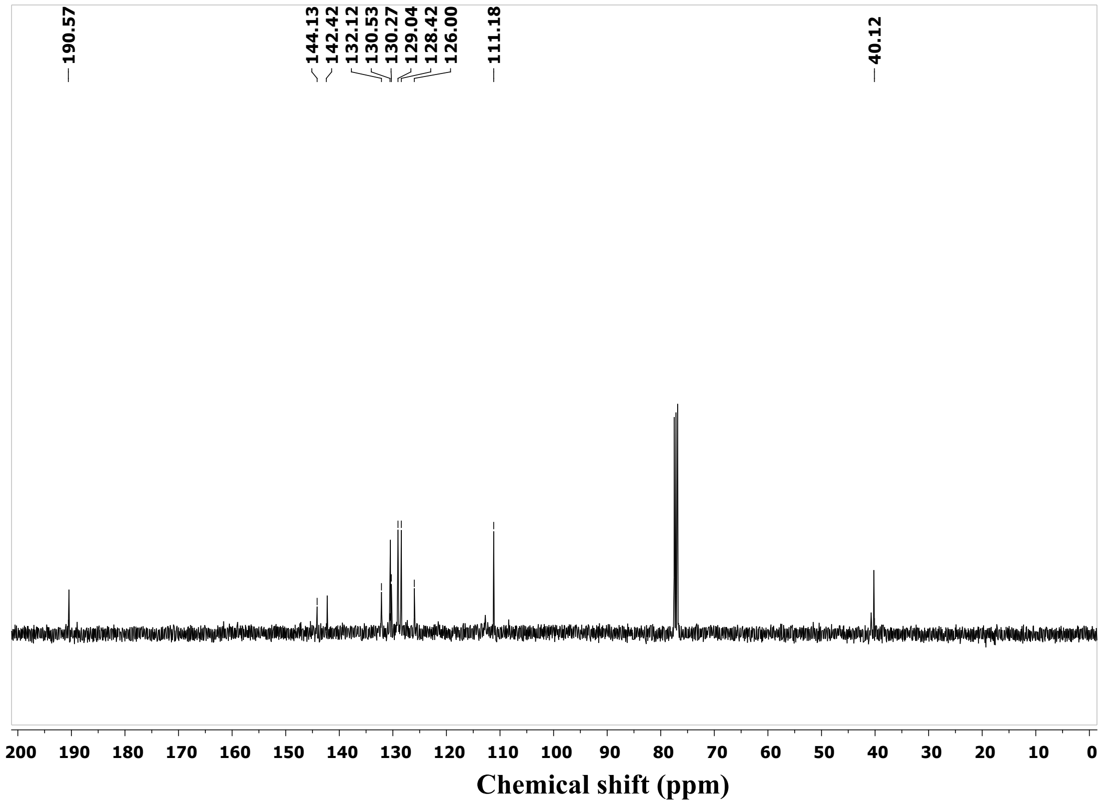




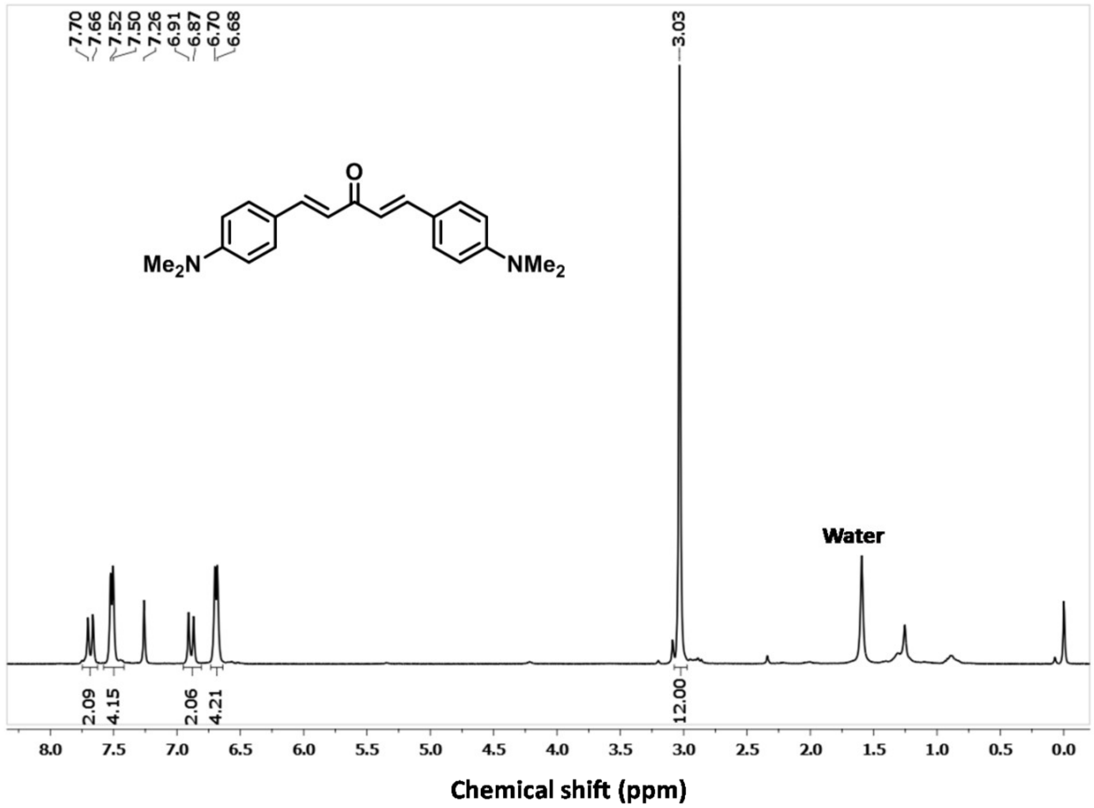
Chemical shift (ppm)

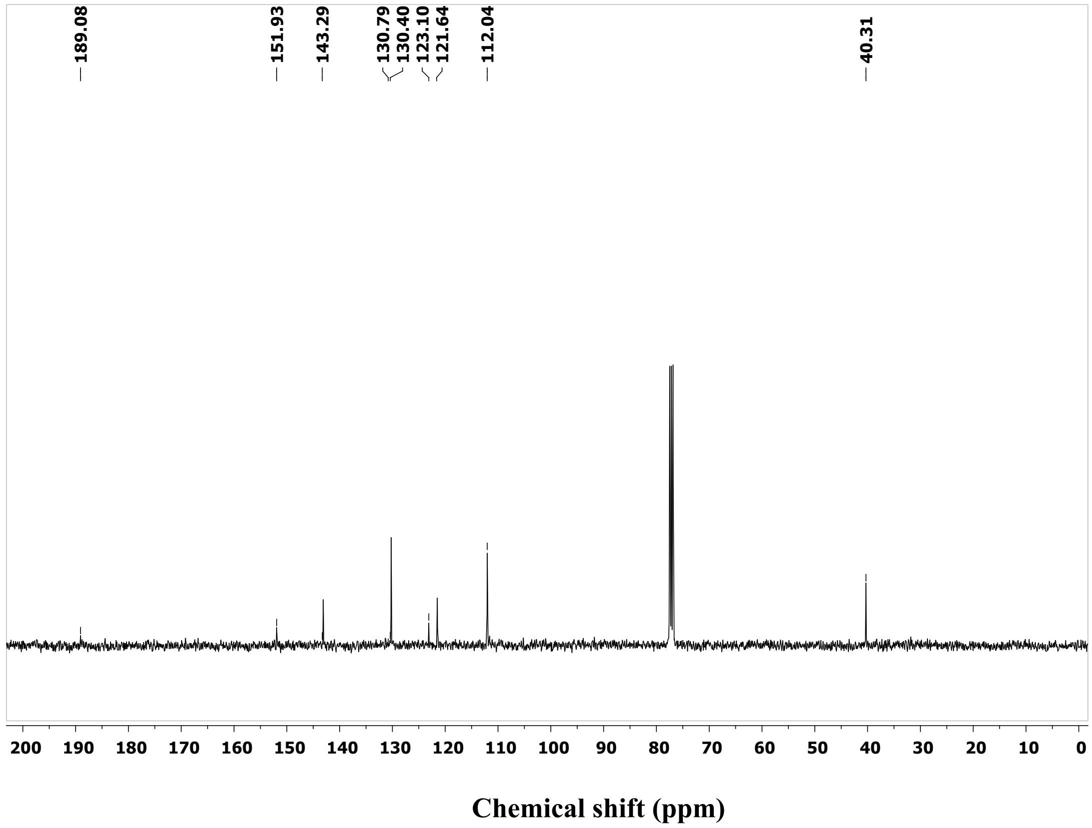
^{1}H and ^{13}CNMR Spectra of DBA-1





^{1}H and ^{13}CNMR Spectra of DBA-2





^{1}H and ^{13}CNMR Spectra of DBA-3

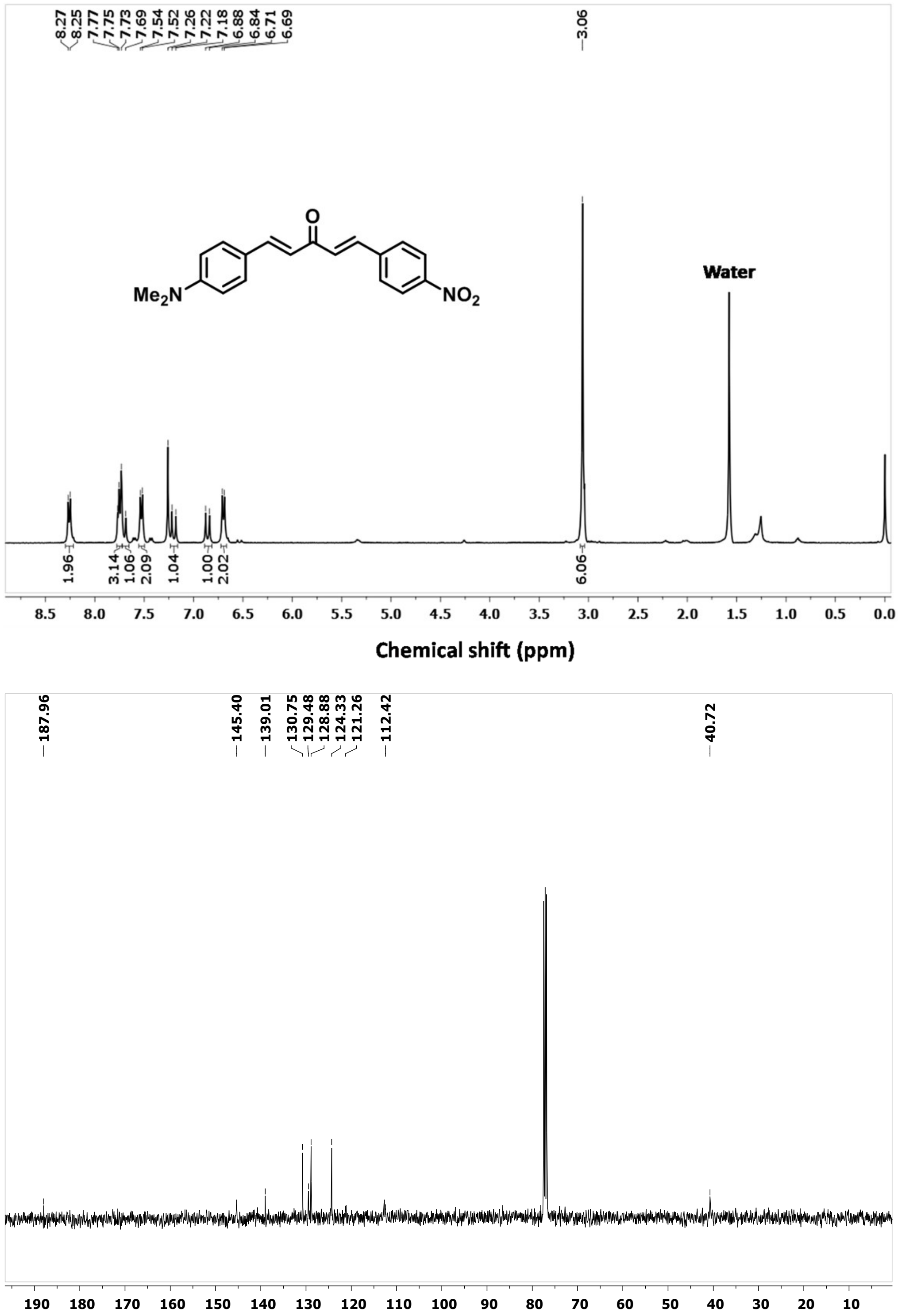


Fig. S1. ^{1}H and ^{13}CNMR Spectra of DBA derivatives (**DBA-DBA-3**)

4. Table S1. Elemental analysis data for the compounds DBA - DBA-3.

S.No	Compound Name	Molecular Formula	% Carbon		% Hydrogen		% Nitrogen	
			Calculated	Found	Calculated	Found	Calculated	Found
1	DBA	$C_{17}H_{14}O$	87.15	85.78	6.02	6.07	-	-
2	DBA-1	$C_{19}H_{19}NO$	82.28	78.62	6.90	6.95	5.05	5.63
3	DBA-2	$C_{21}H_{24}N_2O$	78.71	77.77	7.55	7.65	8.74	8.19
4	DBA-3	$C_{19}H_{18}N_2O_3$	70.79	70.15	5.63	5.76	8.69	8.25

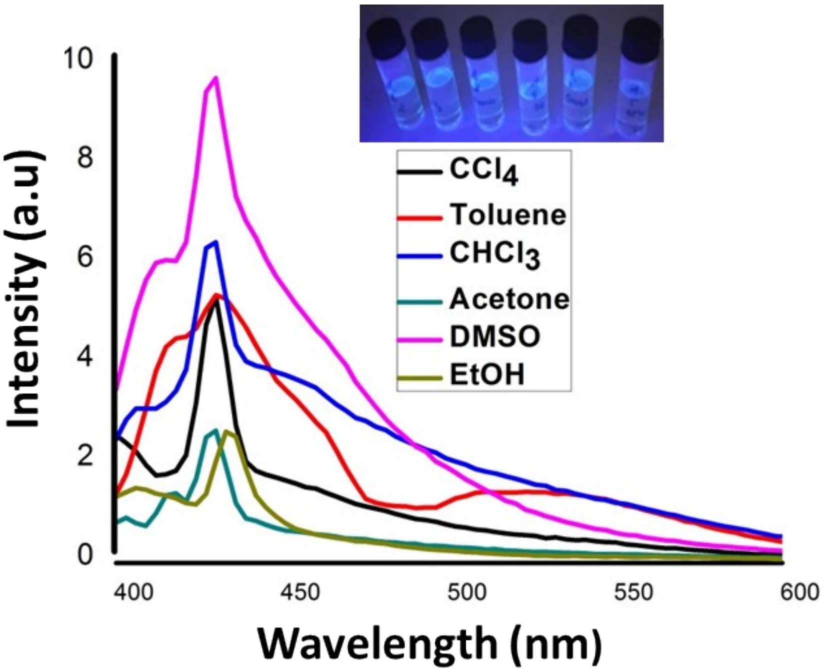


Fig. S2 Emission spectra of **DBA** in different solvents. (380 nm excitation, 5 µM concentration).

5. Calculation of dipole moment change (Δμ) using Mataga-Lippert diagram.

The relationship between stokes shift and solvent polarity was usually demonstrated by Lippert-Mataga equation (eq 1). The change in magnitude of the dipole moment (Δμ) between the ground and excited states can be calculated by using this equation.

$$\Delta v = \frac{2\Delta f}{hca^3}(\Delta\mu)^2 + const \qquad \ldots\ldots(1)$$

From this equation, Δν is the Stokes shift (in wavenumber); Δf is the orientation polarizability of the solvent, h is Planck's constant, and c is the speed of light; a is the radius of the Onsager cavity.

Table S2 and Table S3 displays the calculation of the Stokes shifts value for DBA-1 and DBA-2.

Table S2 photo physical properties of **DBA-1** in various solvents.

Solvent	solvent orientation Polarizability (Δf)	absorbance λ (nm)	emission λ (nm)	absorbance ν (cm-1)	emission ν (cm-1)	stokes shift Δν (cm-1)
CCl_4	0.011	407	474	24570	21097	3473
Toluene	0.013	411	501	24330	19960	4370
chloroform	0.150	417	544	23980	18382	5598
acetone	0.284	422	604	23696	16556	7140
DMSO	0.263	428	600	23364	16666	6698
Ethanol	0.289	434	593	23041	16863	6178

Table S3 photo physical properties of **DBA-2** in various solvents.

Solvent	solvent orientation Polarizability (Δf)	absorbance λ (nm)	emission λ (nm)	absorbance ν (cm-1)	emission ν (cm-1)	stokes shift Δν (cm-1)
CCl_4	0.011	416	471	24038	21231	2807
Toluene	0.013	427	486	23419	20576	2843
chloroform	0.150	435	529	22988	18903	4085
acetone	0.284	443	545	22573	18348	4225
DMSO	0.263	446	571	22421	17513	4908
Ethanol	0.289	454	599	22026	16694	5332

Figure S3 shows the Lippert relationship between the Stokes shifts and solvent polarities.

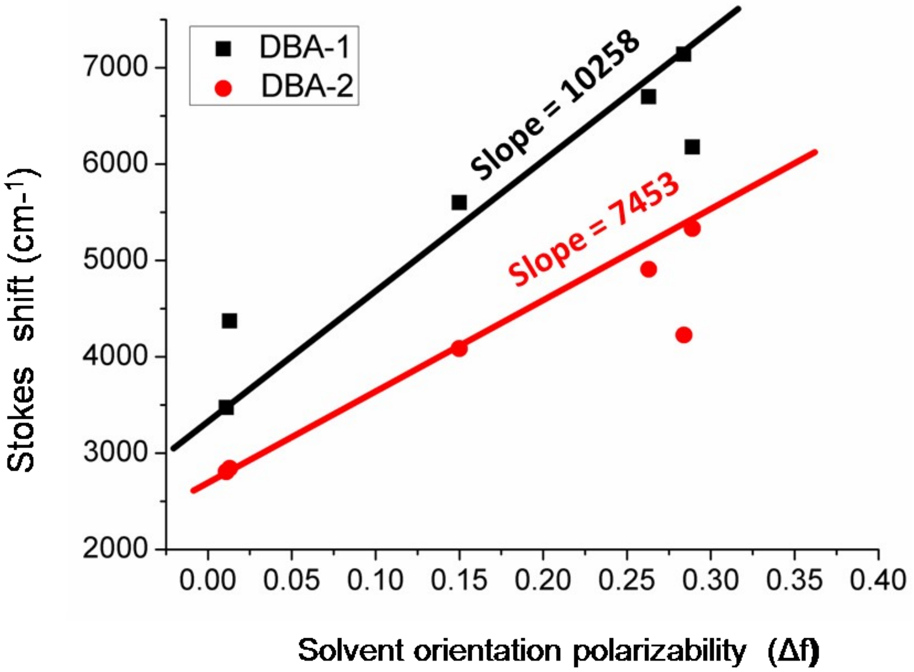


Fig. S3 Lippert–Mataga plot showing Stokes shift vs solvent orientation polarizability (Δf).

The difference between the dipole moment of the excited and ground states (Δμ) can be estimated from the slope of a plot of Δν versus Δ*f by* using (eq 2).

$$slope =- \frac{2\Delta f}{hca^3}(\Delta\mu)^2 \qquad (2)$$

We estimated the Onsager radius (a) for DBA-1(5.60 Å) and DBA-2 (5.77 Å), from quantum chemical calculation by using the density functional MN15/def2-SVP theory. According to the slopes obtained from the fitted results as shown in Figure 3, the difference between the ground state and excited-state dipole moments (Δμ) are found to be 13.38 D for DBA-1 and 11.40 D for DBA-2 respectively. Further, by using density functional MN15/def2-SVP theory the ground state dipole moment of DBA-1 (6.30 D), DBA-2 (5.80 D) was calculated. Therefore, the μ_e values were estimated to be 19.68 D for **DBA-1** and 17.20 D for **DBA-2**.

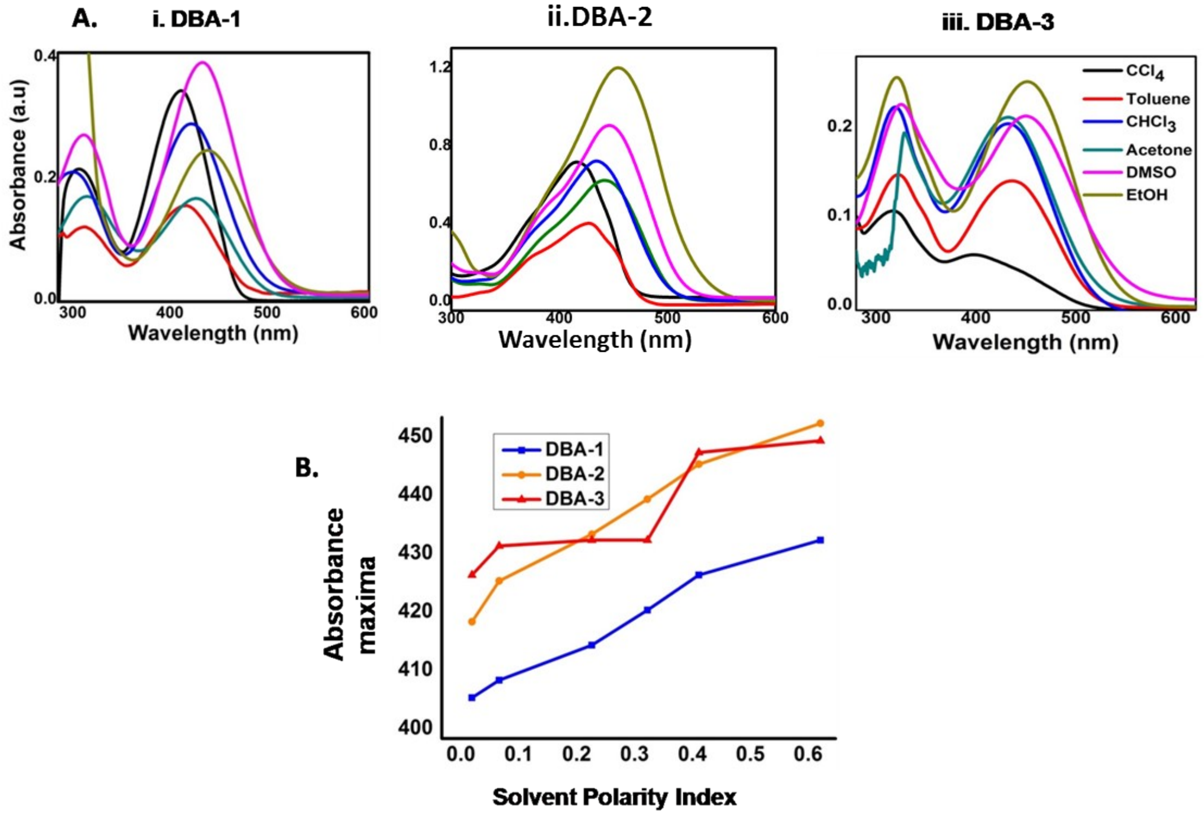
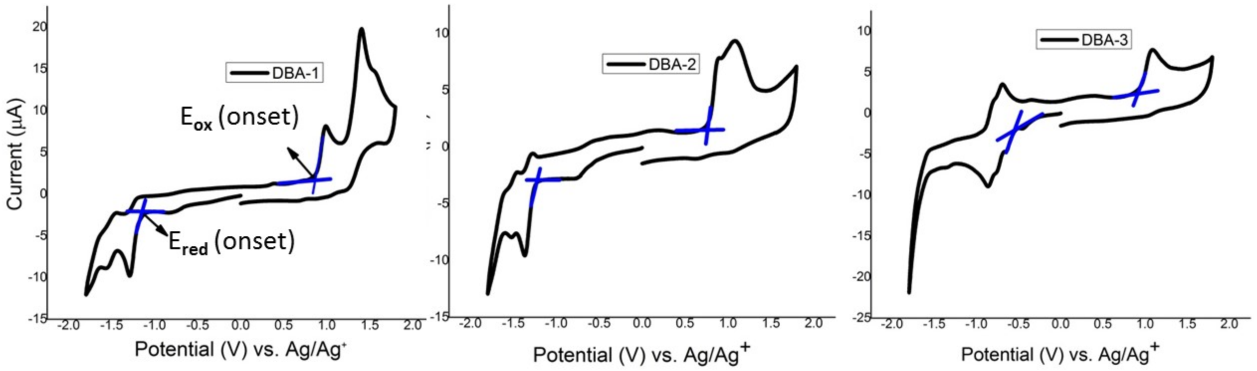


Fig. S4. Absorption spectra of **DBA-1** (i), **DBA-2** (ii) and **DBA-3** (iii) in different solvents. Concentration: 5µM. (B) Plot of absorption maxima VS different polarity of solvents.

6.Cyclic voltammograms of DBA derivatives.

The HOMO and LUMO energy levels of the DBA derivatives (DBA-1-3) were calculated by using cyclic voltammetry (CV). CV experiments were performed on a three-electrode cell system consisting of glassy carbon as a working electrode, platinum as a counter electrode and Ag/Ag^+ as a reference electrode. In this experiment 0.5 mm concentration of DBA derivatives were used in N_2 saturated supporting electrolytes contains 0.1 M tetrabutylammonium perchlorate in DCM solvent. Cyclic voltammetry was recorded in the scan rate of 50 mV/s and the ferrocene redox (Foc/Foc^+) system was used as a known reference for the calculation. Figure 3 shows the cyclic voltammetry profile for DBA derivatives and the oxidation potential (E_{ox}) and reduction potential of the derivatives were obtained from the graph.



E ox (onset) = 0.87 V
E red (onset) = -1.14 V

DBA-1

E_{HOMO}= -(0.87+4.8-0.54)= -5.13 eV

E_{LUMO}= -(-1.14+4.8-0.54)= -3.12 eV

Δ E = 2.01 eV

E ox (onset) = 0.76 V
E red (onset) = -1.22 V

DBA-2

E_{HOMO}= -(0.76+4.8-0.54)= -5.02 eV

E_{LUMO}= -(-1.22+4.8-0.54)= -3.04 eV

Δ E = 1.98 eV

E ox (onset) = 0.91 V
E red (onset) = -0.53 V

DBA-3

E_{HOMO}= -(0.91+4.8-0.54)= -5.17 eV

E_{LUMO}= -(-0.53+4.8-0.54)= -3.73 eV

Δ E = 1.44 eV

Figure S5. Cyclic voltammograms of DBA derivatives.

Further, the highest occupied molecular orbital (HOMO) energy level and the lowest occupied molecular orbital (LUMO) energy level and energy bandgap are calculated by using the following equations:

$$E_{HOMO} = -\ (E\ (ox)\ onset+4.8-E_{FOC})\ (1)$$

$$E_{LUMO} = -\ (E\ (red)\ onset+4.8-E_{FOC})\ (2)$$

$$Eg=-(E_{HOMO}-E_{LUMO})\ (3)$$

E_{HOMO} and E_{LUMO} correspond to the energy levels of HOMO and LUMO, E_{ox} *(onset)* is the onset oxidation potential, *Ered(onset)* is the onset reduction potential, 4.8 is the reference energy of ferrocene (FOC, 4.8 eV under the vacuum level), E_{FOC} is the potential of FOC/FOC+ *versus* Ag/AgCl (0.54 eV, as measured by cyclic voltammetry).

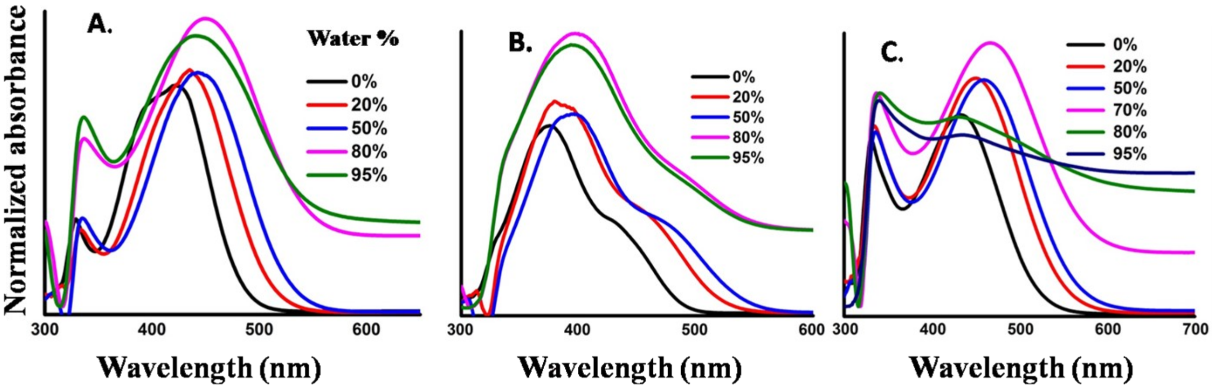
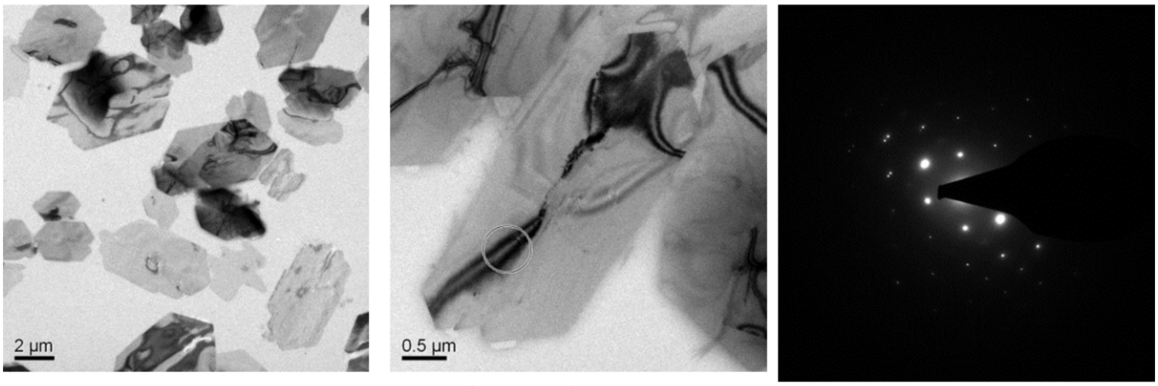


Fig. S6 Absorption spectra of **DBA-1** (a), **DBA-2** (b) and **DBA-3** (c) in acetone – water mixtures with different water contents

A. 60% Water in acetone



B. 95% Water in acetone

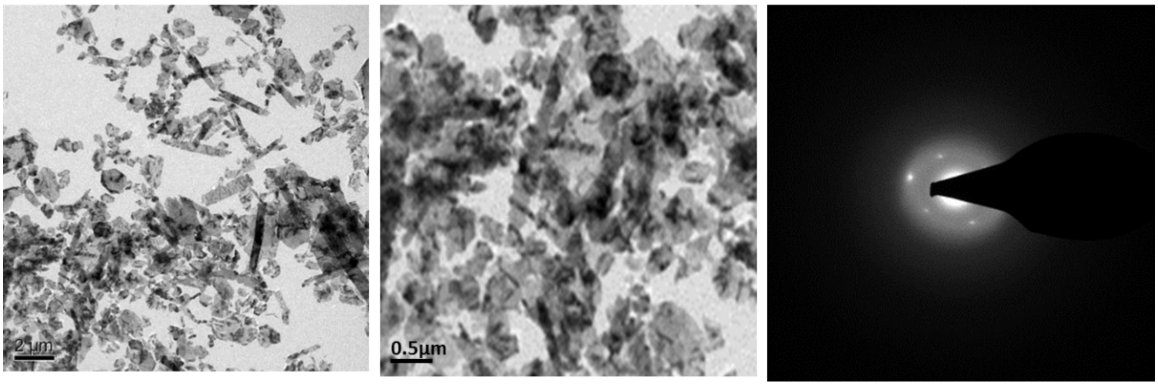


Fig. S7 A. TEM images and ED spectra of DBA-3 aggregates formed in water: acetone mixtures comprising 60% water. **B.** TEM images and ED spectra of DBA-3 aggregates formed in water: acetone mixtures comprising 95% water.

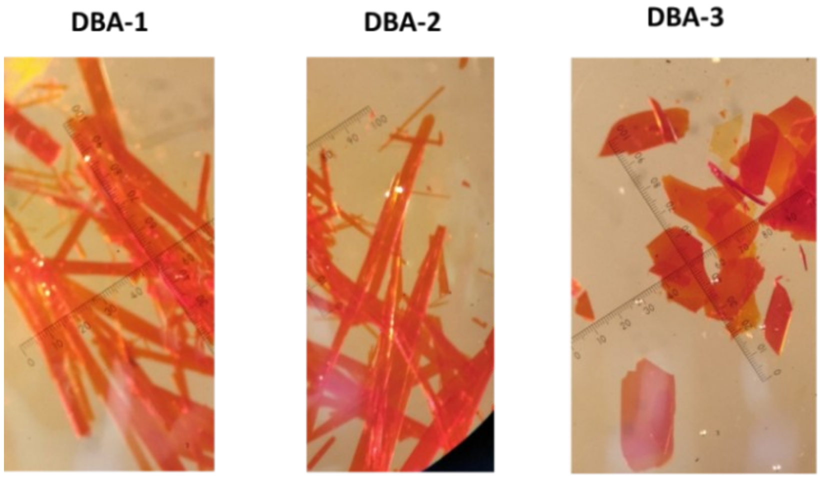


Fig. S8A Single crystals of **DBA- 1, DBA- 2,** and **DBA- 3.**

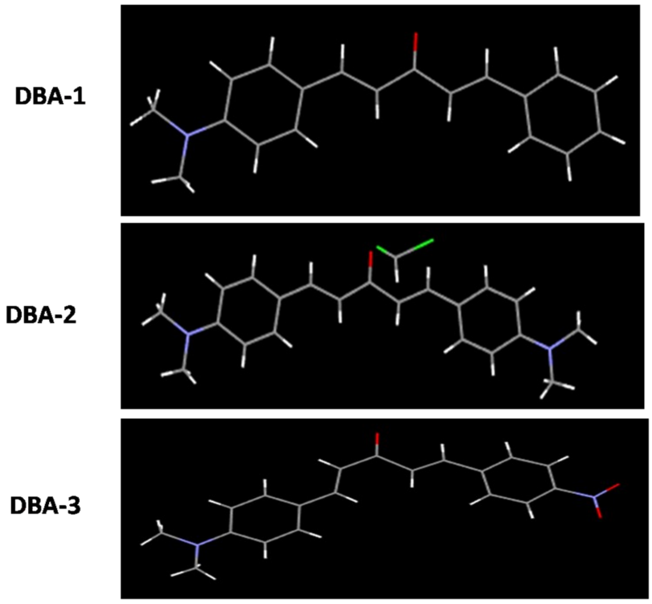


Fig. S8B Single crystal XRD structure of **DBA- 1, DBA- 2,** and **DBA- 3.**

Colors correspond to carbon (gray), hydrogen (white), oxygen (red) and Nitrogen (blue).

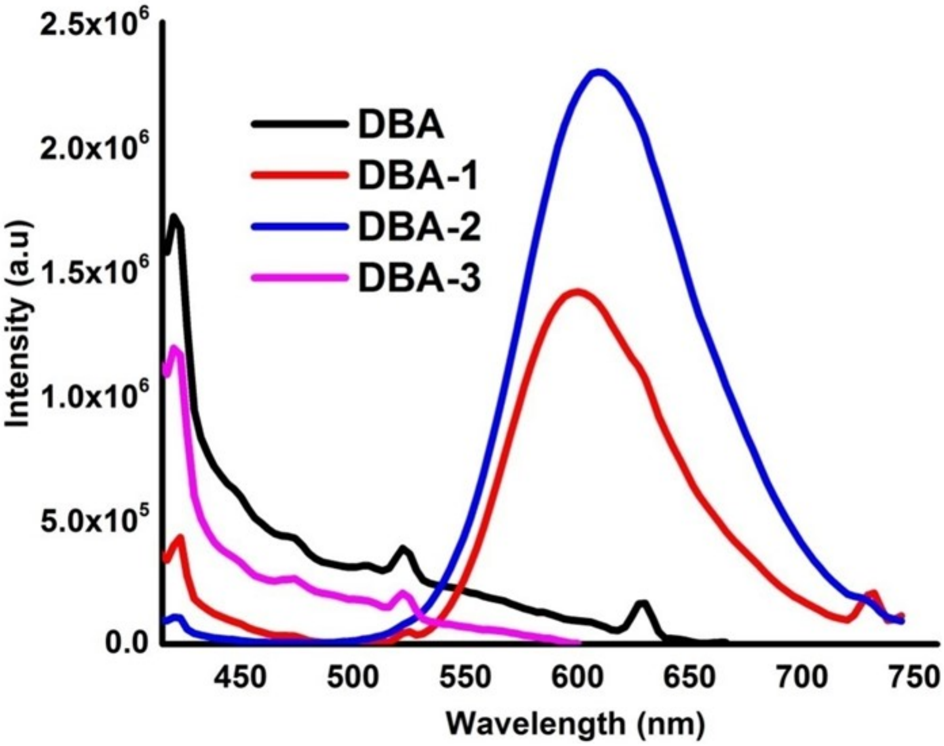


Fig. S9A: Solid state Fluorescence spectra of the **DBA** derivatives in thin films

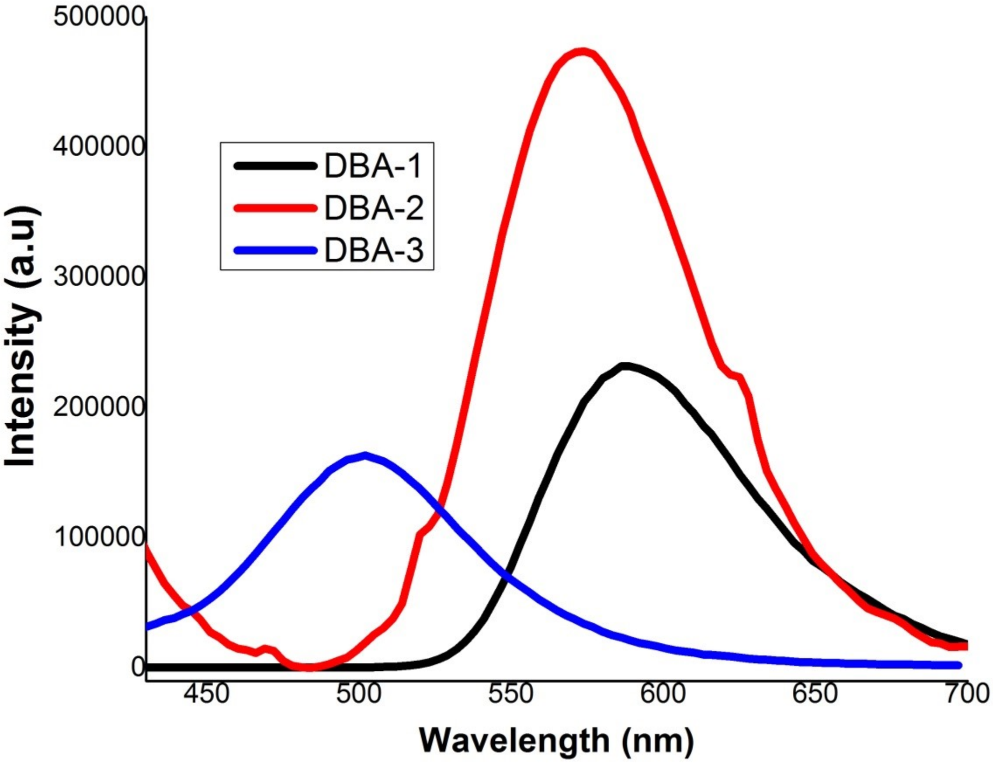


Fig. S9B: Fluorescence spectra of the **DBA** crystals.

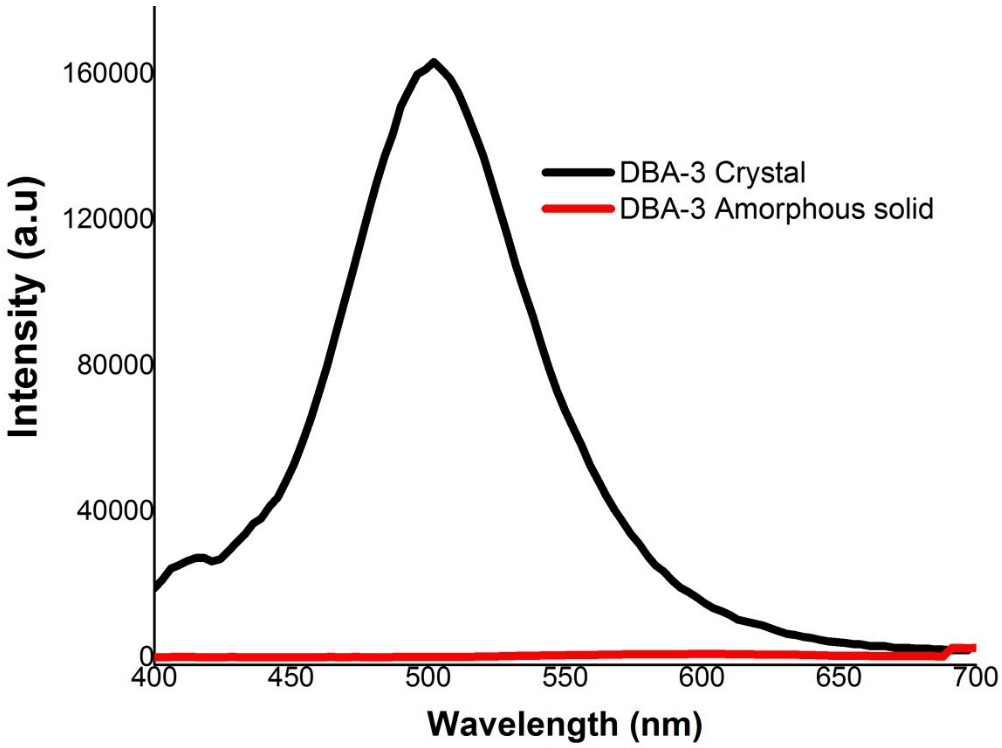


Fig. S9C: Fluorescence spectra of **DBA-3** crystal and amorphous solid.

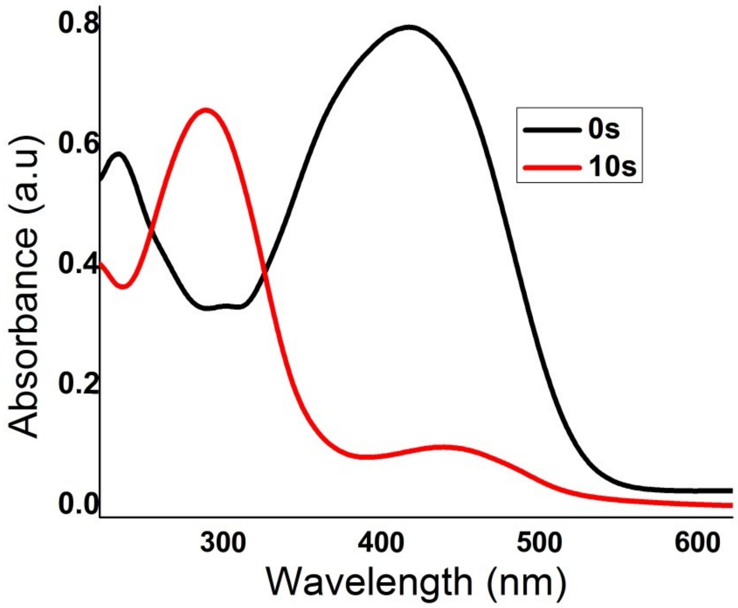


Fig. S10 (i) **DBA-2** film before exposing to saturated DCP vapors (black line) and after exposing to saturated DCP vapors for 10 seconds (red line).

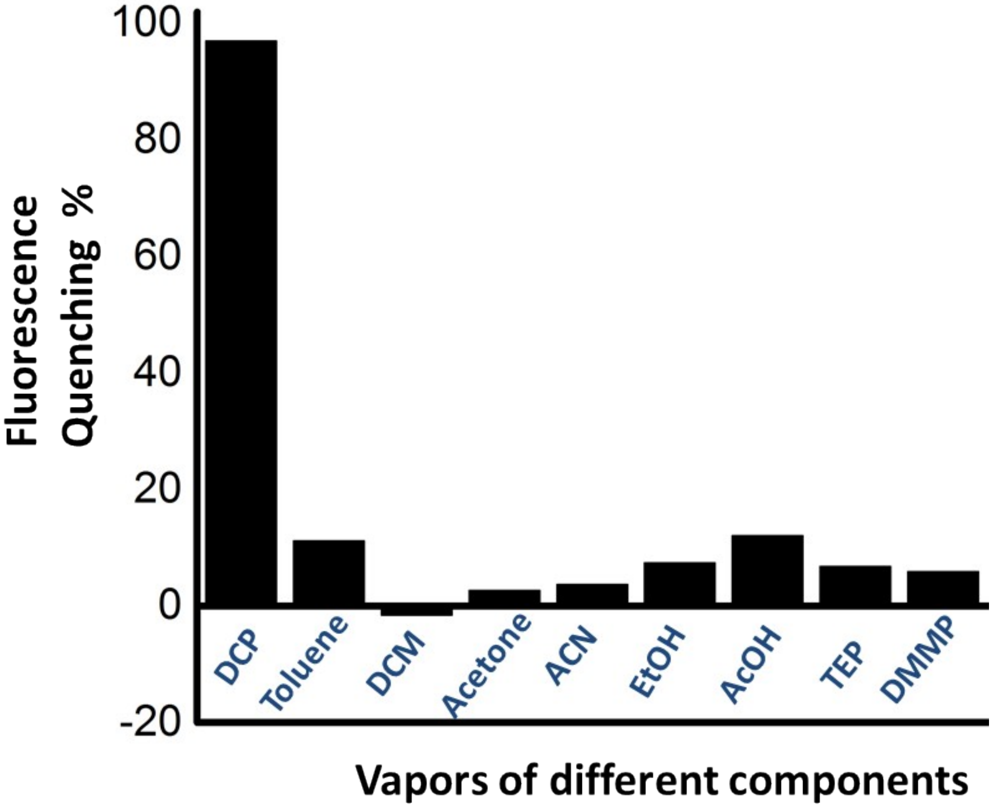


Fig. S11. Fluorescence responses of **DBA-2** to DCP and various common interfering agents.

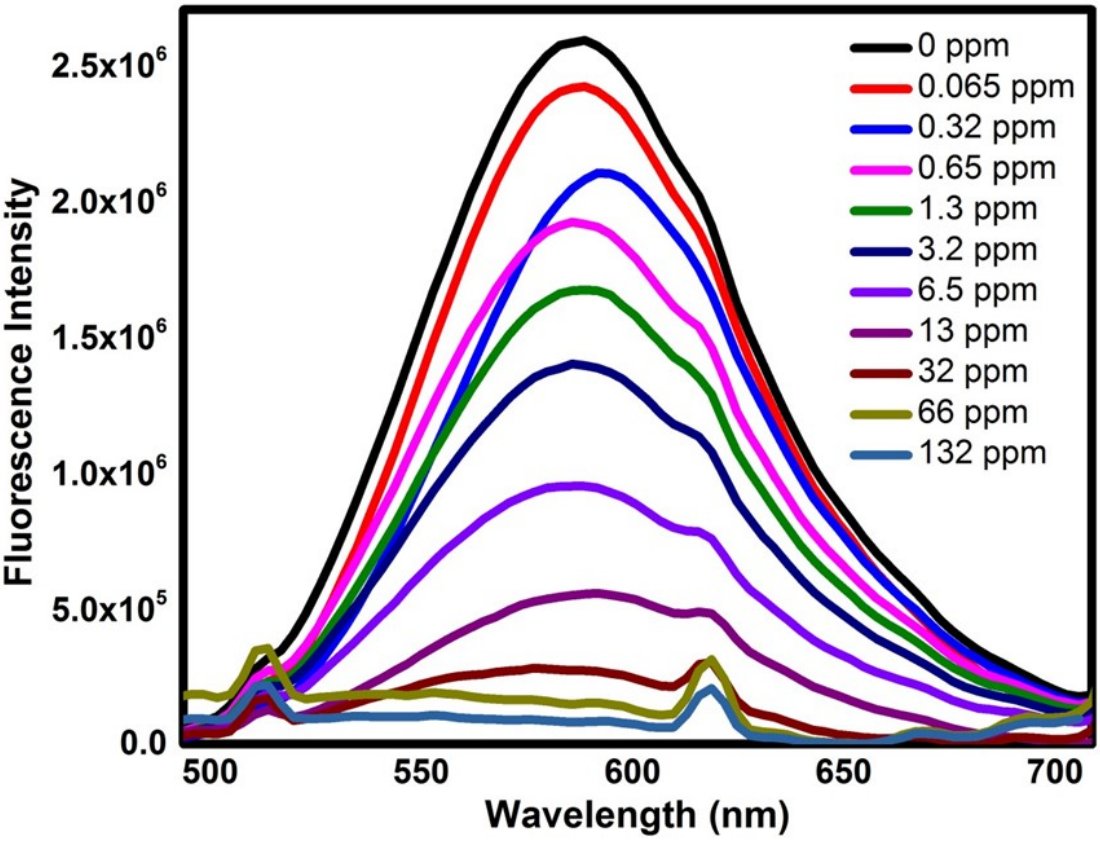


Fig. S12. Fluorescence quenching of **DBA-2** with different concentrations of DCP vapor.

Table S4. Computational Details

Excited state numbers, excitation energies (eV), wavelengths (nm) and oscillator strengths of all the studied compounds in toluene at the MN15/def2-TZVP//MN15-def2-SVP level.

		Toluene		
	Excited state numbers	**Excitation energies (eV)**	**Wavelength (nm)**	**Oscillator strength**
DBA-1	S_1	3.07	404.04	1.2872
	S_2	3.21	386.65	0.0000
	S_3	3.96	312.71	0.4461
	S_4	4.32	286.74	0.0357
	S_5	4.60	269.71	0.1204
	S_6	4.64	267.01	0.0285
	S_7	4.99	248.68	0.0074
	S_8	5.01	247.67	0.1341
	S_9	5.39	230.11	0.0354
	S_{10}	5.55	223.33	0.0988
DBA-2	S_1	3.00	412.64	1.8236
	S_2	3.26	380.16	0.0000
	S_3	3.46	358.02	0.1235
	S_4	4.32	286.94	0.0649
	S_5	4.34	285.72	0.0265
	S_6	4.67	265.36	0.0283
	S_7	4.85	255.62	0.2128
	S_8	5.06	244.92	0.0090
	S_9	5.10	243.14	0.1520
	S_{10}	5.12	242.31	0.0656
DBA-3	S_1	2.81	440.93	1.0225

	S_2	3.11	398.11	0.0000
	S_3	3.73	332.83	0.0000
	S_4	3.76	330.05	0.6769
	S_5	3.88	319.87	0.3342
	S_6	4.34	285.62	0.0255
	S_7	4.38	283.02	0.0260
	S_8	4.72	262.59	0.0076
	S_9	4.76	260.59	0.0456
	S_{10}	4.85	255.68	0.0057

Reference:

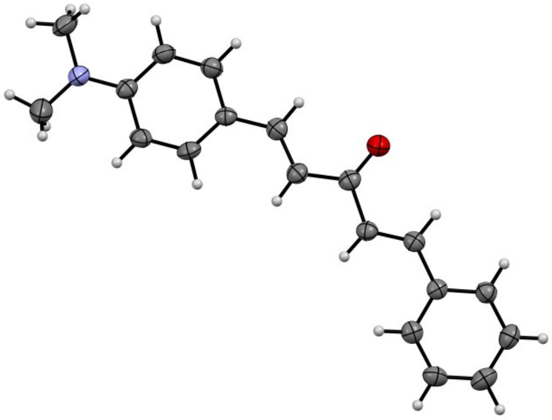
1. M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, R. Kobayashi, N. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G. Zakrzewski, G. A. Voth, P. Salvador, J. J. Dannenberg, S. Dapprich, A. D. Daniels, Ö. Farkas, J. B. Foresman, J. V. Ortiz, J. Cioslowski and D. J. Fox, *Gaussian 16*, Revision A.03; Gaussian, Inc., Wallingford CT, 2016.

Table S5

Crystal data and structure refinement for DBA-1.

Identification code	Manikandan2b
CCDC number	2143170
Empirical formula	$C_{19}H_{19}NO$
Formula weight	277.35
Temperature/K	200.15
Crystal system	orthorhombic
Space group	Pbca
a/Å	11.7069(19)
b/Å	7.5757(12)
c/Å	33.990(5)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3014.5(8)
Z	8
ρ_{calc}g/cm^3	1.222
μ/mm^{-1}	0.075
F(000)	1184.0
Crystal size/mm^3	0.27 × 0.12 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.224 to 49.054
Index ranges	$-13 \le h \le 12$, $-8 \le k \le 8$, $-39 \le l \le 38$
Reflections collected	17541
Independent reflections	2499 [R_{int} = 0.0980, R_{sigma} = 0.0678]
Data/restraints/parameters	2499/0/192
Goodness-of-fit on F^2	1.021
Final R indexes [I>=2σ (I)]	R_1 = 0.0457, wR_2 = 0.0939
Final R indexes [all data]	R_1 = 0.1191, wR_2 = 0.1230
Largest diff. peak/hole / e Å^{-3}	0.15/-0.20

Anisotropic Displacement Parameters of DBA-1



Anisotropic Displacement Parameters ($\AA^2\times10^3$) for DBA-1. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

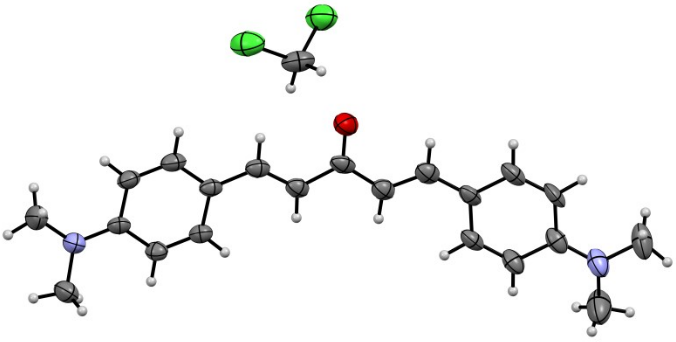
Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O_1	51.0(13)	50.1(11)	41.7(11)	1.2(9)	-4.3(10)	-13.4(11)
N_1	55.3(16)	48.4(13)	36.0(13)	5.9(11)	-12.4(12)	-14.4(12)
C_1	69(2)	60.3(19)	41.4(17)	2.3(15)	-23.0(17)	-6.8(16)
C_2	82(2)	59.0(18)	44.3(18)	7.7(15)	-7.9(17)	-21.7(18)
C_3	37.9(17)	32.6(13)	32.9(15)	-3.0(12)	-2.3(13)	2.1(13)
C_4	38.9(17)	36.4(14)	36.1(15)	-6.0(12)	-1.3(14)	-7.7(13)
C_5	38.9(17)	41.6(15)	32.7(15)	-8.8(12)	-7.8(13)	-1.7(14)
C_6	31.2(16)	30.7(13)	33.5(15)	-5.9(12)	-1.1(13)	0.9(13)
C_7	36.3(17)	39.0(15)	44.1(17)	1.0(13)	-3.2(14)	-3.6(14)
C_8	38.4(18)	44.0(16)	42.3(17)	1.4(13)	-12.9(14)	-2.4(14)
C_9	37.2(17)	36.0(14)	34.6(15)	-5.1(12)	5.3(13)	1.4(13)
C_{10}	40.5(17)	38.3(14)	31.6(15)	-4.7(12)	0.4(14)	-1.2(13)
C_{11}	38.4(17)	33.4(14)	32.9(15)	-6.7(12)	0.7(13)	2.5(14)
C_{12}	36.1(16)	31.6(14)	33.6(15)	-4.6(12)	-0.9(13)	-1.1(12)
C_{13}	35.1(17)	29.6(13)	36.4(16)	-3.0(12)	1.2(13)	1.3(12)
C_{14}	32.4(16)	26.2(12)	32.7(15)	-2.1(11)	-1.1(12)	4.2(13)
C_{15}	35.2(17)	31.5(13)	38.3(16)	-0.8(12)	2.2(13)	4.5(13)
C_{16}	32.1(16)	38.1(15)	49.8(18)	-3.8(14)	-3.8(14)	-0.5(13)
C_{17}	46(2)	48.9(17)	39.2(18)	-6.6(14)	-11.2(15)	9.0(15)
C_{18}	52(2)	47.5(16)	30.1(16)	0.1(13)	0.9(14)	4.3(15)
C_{19}	39.2(18)	35.4(14)	38.7(17)	0.6(12)	0.6(14)	-2.0(13)

Crystal data and structure refinement for DBA-2.

Identification code	**MR_di-NMe2_03_11_20_5**
CCDC number	2143171
Formula	$C_{22}H_{26}Cl_2N_2O$
$D_{calc.}$/ g cm^{-3}	1.269
m/mm^{-1}	2.852

Formula Weight	405.35
Colour	clear orange
Shape	needle
Size/mm^3	0.33×0.06×0.04
T/K	200.00(10)
Crystal System	orthorhombic
Space Group	*Pbca*
a/Å	7.9725(3)
b/Å	46.1355(16)
c/Å	11.5350(3)
a/°	90
b/°	90
g/°	90
V/Å^3	4242.7(2)
Z	8
Z'	1
Wavelength/Å	1.54184
Radiation type	Cu K_a
Q_{min}/°	3.832
Q_{max}/°	68.418
Measured Refl.	15695
Independent Refl.	3733
Reflections with I > 2(I)	2479
R_{int}	0.1655
Parameters	248
Restraints	0
Largest Peak	0.367
Deepest Hole	-0.533
GooF	1.076
wR_2 (all data)	0.3316
wR_2	0.3039
R_1 (all data)	0.1628
R_1	0.1180

Anisotropic Displacement Parameters of DBA-2



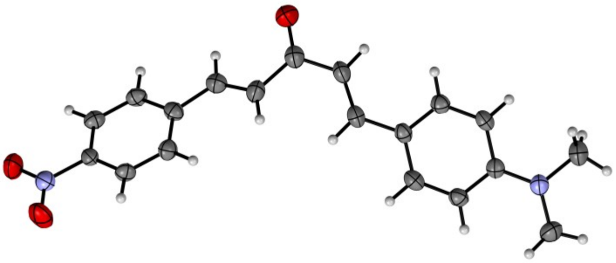
Anisotropic Displacement Parameters ($Å^2×10^3$) for DBA-2. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+…]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	63.6(11)	108.0(16)	60.7(12)	17.4(11)	-3.3(8)	-22.2(11)
Cl2	87.2(14)	93.2(14)	58.2(12)	10.9(10)	-5.6(9)	-22.7(11)
O1	79(3)	48(2)	49(3)	-1(2)	18(2)	10(2)
N1	62(3)	49(3)	37(3)	-2(2)	7(2)	-9(2)
N2	56(3)	59(3)	63(4)	-25(3)	9(3)	0(3)
C3	39(3)	41(3)	29(3)	9(2)	0(2)	2(2)
C9	42(3)	45(3)	34(3)	9(2)	3(2)	8(3)
C8	36(3)	45(3)	35(3)	2(2)	4(2)	-3(2)
C7	34(3)	43(3)	33(3)	8(2)	1(2)	0(2)
C6	36(3)	38(3)	32(3)	9(2)	5(2)	9(2)
C10	45(3)	41(3)	31(3)	4(2)	-1(2)	12(2)
C11	57(3)	46(3)	27(3)	7(2)	2(2)	21(3)
C14	50(3)	47(3)	23(2)	-4(2)	-6(2)	12(3)
C4	49(3)	40(3)	39(3)	10(2)	9(2)	-1(3)
C19	53(3)	51(3)	27(3)	-9(2)	-1(2)	14(3)
C5	46(3)	48(3)	38(3)	12(3)	11(2)	5(3)
C13	52(3)	46(3)	36(3)	3(3)	-4(2)	15(3)
C17	38(3)	54(3)	45(3)	-16(3)	-4(2)	11(3)
C16	68(4)	57(4)	30(3)	-24(3)	-2(3)	9(3)
C18	40(3)	55(3)	44(3)	-16(3)	4(2)	6(3)
C12	50(3)	43(3)	36(3)	2(3)	-1(2)	11(3)
C15	61(4)	60(4)	29(3)	-13(3)	-3(2)	16(3)
C1	84(5)	53(4)	50(4)	-3(3)	1(3)	-20(4)
C2	68(4)	65(4)	37(3)	-6(3)	4(3)	-8(3)
C22	59(4)	79(5)	52(4)	25(4)	8(3)	5(4)
C20	62(4)	60(4)	99(7)	-40(4)	1(4)	-1(4)
C21	91(6)	70(5)	118(8)	-35(5)	41(6)	-19(5)

Crystal data and structure refinement for DBA-3.

Identification code	Manikandan1b
CCDC number	2143172
Empirical formula	$C_{19}H_{18}N_2O_3$
Formula weight	322.35
Temperature/K	200.15
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	5.9990(13)
b/Å	37.205(8)
c/Å	7.1523(16)
α/°	90
β/°	91.421(6)
γ/°	90
Volume/Å^3	1595.8(6)
Z	4
ρ_{calc}g/cm^3	1.342
μ/mm^{-1}	0.092
F(000)	680.0
Crystal size/mm^3	0.24 × 0.15 × 0.09
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.38 to 50.59
Index ranges	$-7 \leq h \leq 7$, $-44 \leq k \leq 42$, $-8 \leq l \leq 8$
Reflections collected	9557
Independent reflections	2657 [R_{int} = 0.0808, R_{sigma} = 0.0944]
Data/restraints/parameters	2657/0/219
Goodness-of-fit on F^2	0.972
Final R indexes [I>=2σ (I)]	R_1 = 0.0562, wR_2 = 0.1054
Final R indexes [all data]	R_1 = 0.1470, wR_2 = 0.1364
Largest diff. peak/hole / e Å^{-3}	0.27/-0.21

Anisotropic Displacement Parameters of DBA-3



Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for DBA-3. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O_1	41.9(14)	45.6(17)	112(2)	4.6(14)	8.5(14)	4.7(12)
O_2	53.5(15)	32.2(16)	103(2)	8.3(15)	-4.6(14)	4.2(13)
O_3	42.9(14)	54.8(18)	84(2)	4.6(14)	-18.6(13)	-13.9(12)
N_1	37.6(14)	29.0(17)	46.5(18)	-0.6(14)	-0.6(12)	-1.2(14)
N_2	37.6(16)	38.0(19)	42.4(18)	-6.4(15)	5.3(13)	-2.5(14)
C_1	36.5(18)	45(2)	77(3)	-9(2)	0.5(18)	7.5(17)
C_2	54(2)	32(2)	65(3)	-0.7(18)	4.2(18)	-3.3(17)
C_3	32.6(16)	29(2)	29.4(19)	0.2(15)	-5.6(14)	-0.2(15)
C_4	32.1(16)	30(2)	33(2)	3.7(15)	-2.9(14)	-8.0(15)
C_5	31.0(16)	34(2)	32(2)	-2.3(15)	-0.6(13)	-2.0(15)
C_6	29.8(16)	28(2)	34(2)	1.7(15)	-5.7(14)	-3.8(14)
C_7	33.1(17)	37(2)	38(2)	1.7(16)	-0.4(14)	-8.4(16)
C_8	26.9(15)	37(2)	37(2)	-2.1(16)	-1.1(14)	-2.6(15)
C_9	39.9(18)	28(2)	40(2)	-0.9(16)	-1.0(15)	-7.2(15)
C_{10}	42.0(19)	28(2)	56(2)	2.4(17)	0.0(17)	-8.4(17)
C_{11}	45(2)	33(2)	58(2)	1.0(18)	0.6(17)	-2.6(17)
C_{12}	43(2)	30(2)	62(3)	-5.3(18)	-1.4(17)	4.1(17)
C_{13}	34.3(17)	38(2)	50(2)	-0.4(18)	-0.3(15)	-0.8(17)
C_{14}	32.5(16)	36(2)	30.4(19)	0.3(15)	2.2(14)	-3.3(15)
C_{15}	27.4(15)	38(2)	34(2)	0.3(16)	-4.7(13)	2.7(16)
C_{16}	31.5(16)	37(2)	33(2)	1.5(15)	0.8(14)	4.5(15)
C_{17}	28.5(15)	28(2)	33(2)	-2.8(15)	4.2(13)	-2.8(15)
C_{18}	25.4(15)	37(2)	38(2)	-0.8(16)	-7.1(14)	2.5(15)
C_{19}	37.0(17)	32(2)	39(2)	4.5(16)	0.6(15)	2.6(16)

Reference:

Mercury Software from CCDC: http://www.ccdc.cam.ac.uk/Solutions/CSDSystem/Pages/Mercury.aspx.