Supporting Information

for

The C–I···$^{-}$O–N^{+} Halogen Bonds with Tetraiodoethylene and Aromatic N-oxides

Khai-Nghi Truong [1], J. Mikko Rautiainen [1], Kari Rissanen [1], and Rakesh Puttreddy [1,2]*
[1]Department of Chemistry, University of Jyvaskyla, P. O. Box 35, Jyväskylä, FI-40014, Finland.
[2]Faculty of Engineering and Natural Sciences, Tampere University, P. O. Box 541, Tampere, FI-33101, Finland.

Table of Contents

1. General Information

All solvents employed for synthesis and crystallization experiments were commercially purchased and were used as received without any purification. In addition, the following chemicals were used as purchased without further purification: Pyridine *N*-oxide (95 %, Sigma Aldrich), 2-picoline *N*-oxide (≥ 96 %, Sigma Aldrich), 3-picoline *N*-oxide (98 %, Sigma Aldrich), 4-picoline *N*-oxide (98 %, Sigma Aldrich), 4-phenylpyridine *N*-oxide (98 %, Sigma Aldrich), and tetraiodoethylene (97 %, Sigma Aldrich).

2. Synthesis and Crystallization Experiments

Aromatic *N*-oxides, **1**-**4**, **10**, and tetraiodoethylene (C_2I_4) were purchased from Sigma Aldrich. Aromatic *N*-oxides, **5**, **6**, **7**, **8**, **9**, and **11**-**13** were synthesized by oxidation of their corresponding *N*-heterocyclic compounds using procedures as reported by Katritzky and Lagowski.[1]

For crystallization of the halogen bonded complexes, a 4 mL vial is charged with **1 - 13** (0.019 mmol, 1 equiv.), respectively, tetraiodoethylene (10 mg, 0.019 mmol, 1 equiv.), and a solvent mixture of DCM/MeOH (*v*:*v*, 1:1, 2 mL) at room temperature under air. The components were stirred at 60 °C for 30 min, and slow evaporation of the resultant yellowish solution at ambient temperature gave single crystals suitable for X-ray diffraction analysis after 3 hours to 4 days. The analogous reaction with **11** – **13** · C_2I_4 did not lead to the desired complex formation; brownish oil or crystals of the starting materials (for example, CSD-Refcode: RAJGUG, AQOZUD) were observed.

3. X-ray Crystallography

All data were measured using (a) a dual-source Rigaku SuperNova diffractometer equipped with an Atlas detector and an Oxford Cryostream cooling system using mirror-monochromated Mo-K_α radiation (λ = 0.71073 Å). Data collection and reduction for all complexes were performed using the program *CrysAlisPro*[2] and Gaussian face-index absorption correction method was applied;[2] (b) a Bruker-Nonius KappaCCD diffractometer with an APEX-II detector with graphite-monochromatized Mo-K_α (λ = 0.71073 Å) radiation. Data collection and reduction were performed using the program *COLLECT*[3] and *HKL DENZO AND SCALEPACK*,[4] respectively, and the intensities were corrected for absorption using *SADABS*.[5] All structures were solved with Direct Methods or Patterson synthesis (*SHELXS*)[6] and refined by full-matrix least squares based on F^2 using *SHELXL*-2013.[6] Non-hydrogen atoms were assigned anisotropic displacement parameters unless stated otherwise. Hydrogen atoms were placed in idealized positions and included as riding. Isotropic displacement parameters for all H atoms were constrained to multiples of the equivalent displacement parameters of their parent atoms with $U_{iso}(H) = 1.2\ U_{eq}$ (parent atom). For a few reported structures, several reflections with large discrepancies between the calculated and observed structure factors have been omitted from the least-squares refinement as outliers. In addition, enhanced rigid bond restraints (RIGU)[7] with standard uncertainties of 0.001 Å^2 were applied for several atom pairs as well as distance restraints (DFIX). Positional disorders were refined to the respective two split positions (see cifs), with the sum of the site occupancies of both alternative positions constrained to unity. Crystals of **2** · C_2I_4 and **5** · C_2I_4 proved to be non-merohedral twins. In the twin refinement, an approximately modified set of intensity data taking the partially overlapped diffraction of a two-component twin into account (HKLF5 format in SHELXL-2015)[8] gave significantly improved convergence results. The X-ray single crystal data and experimental details and CCDC numbers (1992629-1992638) are given below.

Crystal data for **1** · 0.5C_2I_4: CCDC-1992635, $C_6H_5I_2NO$, M = 360.91 gmol^{-1}, colourless block, 0.34 × 0.26 × 0.25 mm^3, triclinic, space group *P*-1 (No. 2), a = 7.6927(5) Å, b = 8.3221(6) Å, c = 8.3398(7) Å, α = 66.0800(10)°, β = 82.897(2)°, γ = 65.7200(10)°, V = 444.29(6) Å^3, Z = 2, D_{calc} = 2.698 gcm^{-3}, F(000) = 324, μ = 7.015 mm^{-1}, T = 120(2) K, θ_{max} = 29.2°, 3315 total reflections, 1602 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0257, 1806 data, 91 parameters, 0 restraints, GooF = 1.034, R_1 = 0.0197 and wR_2 = 0.0410 [$I_o > 2\sigma(I_o)$], R_1 = 0.0253 and wR_2 = 0.0433 (all reflections), 0.790 < d$\Delta\rho$ < -0.916 eÅ^{-3}.

Crystal data for **2** · 0.5C_2I_4: CCDC-1992633, $C_7H_7I_2NO$, M = 374.94 gmol^{-1}, colourless plate, 0.21 × 0.20 × 0.08 mm^3, triclinic, space group *P*-1 (No. 2), a = 7.9003(7) Å, b = 8.1553(8) Å, c = 8.8661(7) Å, α = 66.2800(10)°, β = 76.870(2)°, γ = 84.544(2)°, V = 509.30(8) Å^3, Z = 2, D_{calc} = 2.445 gcm^{-3}, F(000) = 340, μ = 6.125 mm^{-1}, T = 120(2) K, θ_{max} = 28.4°, 3383 total reflections, 1840 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0441, 2047 data, 102 parameters, 0 restraints, GooF = 1.079, R_1 = 0.0335 and wR_2 = 0.0921 [$I_o > 2\sigma(I_o)$], R_1 = 0.0374 and wR_2 = 0.0955 (all reflections), 1.991 < d$\Delta\rho$ < -1.138 eÅ^{-3} (before twin refinement: GooF = 1.563, R_1 = 0.0503, wR_2 = 0.2040, 3.828 < d$\Delta\rho$ < -1.298 eÅ^{-3}).

Crystal data for **3** · 0.5C_2I_4: CCDC-1992629, $C_7H_7I_2NO$, M = 374.94 gmol^{-1}, colourless block, 0.33 × 0.30 × 0.30 mm^3, triclinic, space group *P*-1 (No. 2), a = 8.2502(6) Å, b = 8.2782(5) Å, c = 8.6390(6) Å, α = 80.745(2)°, β = 69.6240(10)°, γ = 64.4300(10)°, V = 498.89(6) Å^3, Z =

2, D_{calc} = 2.496 gcm^{-3}, F(000) = 340, μ = 6.253 mm^{-1}, T = 120(2) K, θ_{max} = 29.3°, 3591 total reflections, 1751 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0231, 2009 data, 101 parameters, 0 restraints, GooF = 1.011, R_1 = 0.0231 and wR_2 = 0.0425 [$I_o > 2\sigma(I_o)$], R_1 = 0.0280 and wR_2 = 0.0460 (all reflections), 1.173 < dΔρ < -0.873 eÅ^{-3}.

Crystal data for **4**·0.5C_2I_4: CCDC-1992638, $C_7H_7I_2NO$, M = 374.94 gmol^{-1}, colourless block, 0.27 × 0.25 × 0.25 mm^3, triclinic, space group *P*-1 (No. 2), a = 7.7326(4) Å, b = 8.3564(5) Å, c = 8.8039(6) Å, α = 107.725(2)°, β = 109.920(2)°, γ = 96.9090(10)°, V = 493.26(5) Å^3, Z = 2, D_{calc} = 2.524 gcm^{-3}, F(000) = 340, μ = 6.324 mm^{-1}, T = 120(2) K, θ_{max} = 29.1°, 3548 total reflections, 1761 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0207, 1994 data, 101 parameters, 0 restraints, GooF = 1.046, R_1 = 0.0223 and wR_2 = 0.0414 [$I_o > 2\sigma(I_o)$], R_1 = 0.0272 and wR_2 = 0.0442 (all reflections), 1.167 < dΔρ < -0.892 eÅ^{-3}.

Crystal data for **5**·0.5C_2I_4: CCDC-1992631, $C_8H_9I_2NO$, M = 388.96 gmol^{-1}, colourless rod, 0.36 × 0.22 × 0.18 mm^3, triclinic, space group *P*-1 (No. 2), a = 7.3902(5) Å, b = 7.6046(4) Å, c = 10.3440(7) Å, α = 105.646(2)°, β = 93.6550(10)°, γ = 92.7480(10)°, V = 557.36(6) Å^3, Z = 2, D_{calc} = 2.318 gcm^{-3}, F(000) = 356, μ = 5.601 mm^{-1}, T = 120(2) K, θ_{max} = 27.0°, 6694 total reflections, 3333 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0571, 4450 data, 122 parameters, 12 restraints, GooF = 1.060, R_1 = 0.0295 and wR_2 = 0.0419 [$I_o > 2\sigma(I_o)$], R_1 = 0.0456 and wR_2 = 0.0429 (all reflections), 1.281 < dΔρ < -0.573 eÅ^{-3}.

Crystal data for 2(**6**)·1.5C_2I_4: CCDC-1992636, $C_{17}H_{18}I_6N_2O_2$, M = 1043.73 gmol^{-1}, colourless block, 0.27 × 0.26 × 0.24 mm^3, monoclinic, space group $P2_1/n$ (No. 14), a = 8.3737(3) Å, b = 34.0685(14) Å, c = 9.8207(4) Å, α = 90°, β = 109.620(2)°, γ = 90°, V = 2638.98(18) Å^3, Z = 4, D_{calc} = 2.627 gcm^{-3}, F(000) = 1872, μ = 7.077 mm^{-1}, T = 170(2) K, θ_{max} = 28.7°, 24357 total reflections, 3723 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0820, 5332 data, 248 parameters, 6 restraints, GooF = 1.034, R_1 = 0.0521 and wR_2 = 0.1016 [$I_o > 2\sigma(I_o)$], R_1 = 0.0787 and wR_2 = 0.0521 (all reflections), 1.160 < dΔρ < -1.301 eÅ^{-3}.

Crystal data for 2(**7**)·C_2I_4: CCDC-1992637, $C_{18}H_{22}I_4N_2O_2$, M = 805.97 gmol^{-1}, colourless plate, 0.43 × 0.31 × 0.13 mm^3, orthorhombic, space group *Pbcn* (No. 60), a = 20.0041(8) Å, b = 7.7412(2) Å, c = 15.5738(7) Å, α = 90°, β = 90°, γ = 90°, V = 2411.69(16) Å^3, Z = 4, D_{calc} = 2.220 gcm^{-3}, F(000) = 1488, μ = 5.182 mm^{-1}, T = 170(2) K, θ_{max} = 29.1°, 14226 total reflections, 1676 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0587, 2435 data, 132 parameters, 6 restraints, GooF = 1.041, R_1 = 0.0414 and wR_2 = 0.0782 [$I_o > 2\sigma(I_o)$], R_1 = 0.0704 and wR_2 = 0.0863 (all reflections), 0.605 < dΔρ < -0.687 eÅ^{-3}.

Crystal data for **8**·0.5C_2I_4: CCDC-1992630, $C_{12}H_9I_2NO$, M = 437.00 gmol^{-1}, colourless plate, 0.24 × 0.20 × 0.07 mm^3, monoclinic, space group *I*2/*a* (No. 15), a = 21.1361(6) Å, b = 7.5995(2) Å, c = 15.8000(5) Å, α = 90°, β = 94.700(2)°, γ = 90°, V = 2529.32(13) Å^3, Z = 8, D_{calc} = 2.295 gcm^{-3}, F(000) = 1616, μ = 4.952 mm^{-1}, T = 120(2) K, θ_{max} = 28.8°, 4879 total reflections, 2026 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0320, 2562 data, 156 parameters, 15 restraints, GooF = 1.002, R_1 = 0.0302 and wR_2 = 0.0536 [$I_o > 2\sigma(I_o)$], R_1 = 0.0406 and wR_2 = 0.0607 (all reflections), 0.746 < dΔρ < -0.852 eÅ^{-3}.

Crystal data for **9**·C_2I_4: CCDC-1992632, $C_{13}H_9I_4NO$, M = 702.81 gmol^{-1}, colourless plate, 0.37 × 0.30 × 0.08 mm^3, triclinic, space group *P*-1 (No. 2), a = 7.5426(3) Å, b = 10.9954(7) Å, c = 20.5768(12) Å, α = 76.4240(10)°, β = 83.242(2)°, γ = 87.166(2)°, V = 1646.89(16) Å^3, Z = 4, D_{calc} = 2.835 gcm^{-3}, F(000) = 1256, μ = 7.560 mm^{-1}, T = 120(2) K, θ_{max} = 28.3°, 12555 total reflections, 4471 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0424, 6661 data, 350 parameters, 59 restraints, GooF = 1.029, R_1 = 0.0457 and wR_2 = 0.0777 [$I_o > 2\sigma(I_o)$], R_1 = 0.0804 and wR_2 = 0.0971 (all reflections), 2.123 < dΔρ < -1.439 eÅ^{-3}.

Crystal data for 2(**10**) · C_2I_4: CCDC-1992634, $C_{24}H_{18}I_4N_2O_2$, M = 874.00 gmol^{-1}, colourless plate, 0.36 × 0.19 × 0.07 mm^3, monoclinic, space group *C*2 (No. 5), a = 33.7738(8) Å, b = 7.5548(2) Å, c = 19.8264(7) Å, α = 90°, β = 90.413(2)°, γ = 90°, V = 5058.7(3) Å^3, Z = 8, D_{calc} = 2.295 gcm^{-3}, F(000) = 3232, μ = 4.952 mm^{-1}, T = 120(2) K, θ_{max} = 29.2°, 30592 total reflections, 8096 with $I_o > 2\sigma(I_o)$, R_{int} = 0.0401, 10195 data, 616 parameters, 86 restraints, Flack parameter x = 0.01(5), GooF = 1.019, R_1 = 0.0421 and wR_2 = 0.0874 [$I_o > 2\sigma(I_o)$], R_1 = 0.0553 and wR_2 = 0.0953 (all reflections), 0.936 < dΔρ < -0.955 eÅ^{-3}.

4. Solid-State Analyses

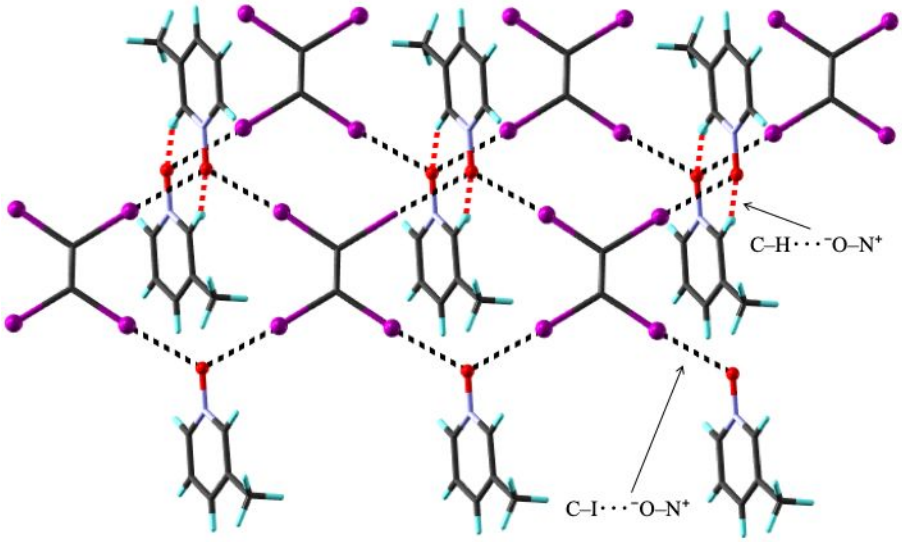

Figure S1. X-ray crystal structure packing of **3** · C_2I_4. The black-dotted lines are C–I···$^-$O–N$^+$ XBs and red-dotted lines represent C–H···$^-$O–N$^+$ interactions.

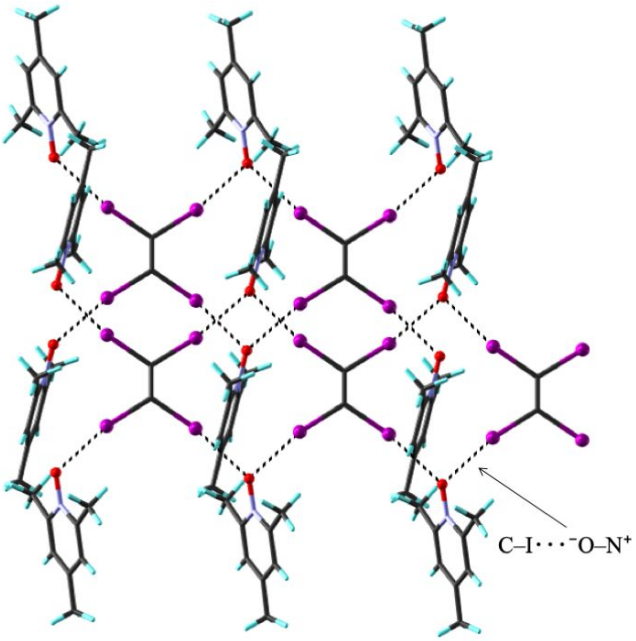

Figure S2. X-ray crystal structure packing of **7** · C_2I_4. The black-dotted lines are C–I···$^-$O–N$^+$ XBs.

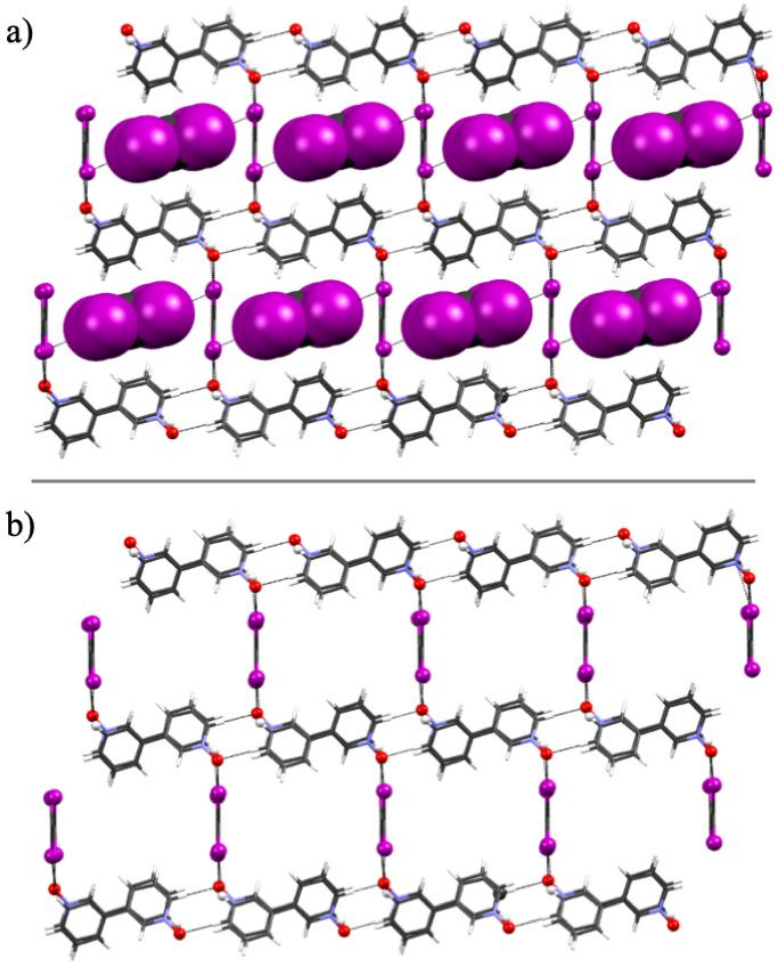

Figure S3. X-ray crystal structure packing of (a) **9**·C_2I_4 and (b) **9**·C_2I_4 with omitted C_2I_4 to show 'porous-like' packing.

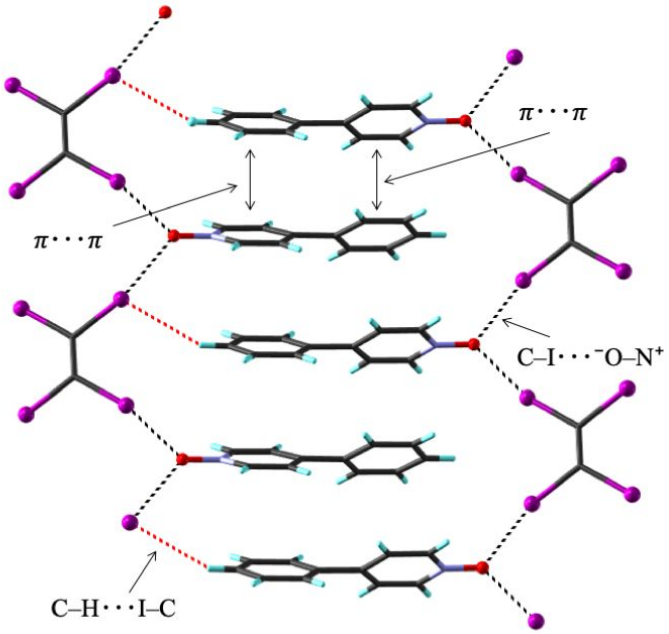

Figure S4. X-ray crystal structure packing of **10**·C_2I_4. The black-dotted lines are C–I···$^{-}$O–N^{+} XBs and red-dotted lines represent C–H···I–C interactions.

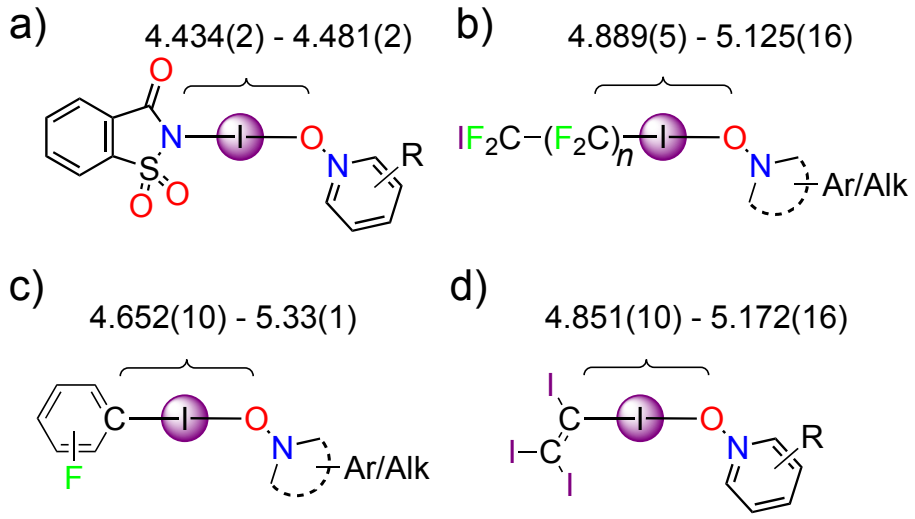

Figure S5. In a D–X···A halogen bonding interaction, comparison of D···A distances [Å] in complexes; (a) (*N*-iodosaccharin)N···O(PyNO) [Type a], (b) (1,ω-perfluoroiodoalkanes)C···O(PyNO) [Type b], (c) (perfluoroiodoarenes)C···O(PyNO) [Type c], and (d) (C_2I_4)C···O(PyNO) [Type d]. Note: PyNOs and XB-donors are monodentate in Type a, and polydentate in Type b, c, and d. Therefore, all C···O distances were considered for comparison purposes.

Table S1: Halogen bond parameters between perfluorohalobenzenes and aromatic/aliphatic N-oxides.

S.No	CSD-Refcode	*d*(I···O) [Å]	∠(C–I···O) [°]	*d*(C···O) [Å]
1	AFUHAN	2.6618(13)	176.17(11)	4.757(4)
2	AFUHER	2.6777(14)	176.40(12)	4.765(4)
3	AFUHIV	2.6900(13)	176.22(11)	4.782(3)
4	COJJOB	2.823(5)	173.90(12)	4.921(7)
5	DAWQUQ	2.784(6)	173.3(2)	4.847(10)
		2.732(6)	174.4(2)	4.809(10)
		2.793(6)	174.9(3)	4.865(10)
		2.806(10)	174.4(3)	4.873(10)
6	DAWRAX	2.728(8)	174.0(2)	4.818(12)
		2.713(3)	175.2(3)	4.788(12)
7	DAWREB	2.771(7)	176.3(3)	4.843(13)
		2.780(7)	177.2(3)	4.861(12)
8	DAWRIF	2.791(5)	172.8(2)	4.871(8)
		2.828(5)	175.3(2)	4.923(8)
		2.800(5)	175.5(2)	4.877(9)
		2.765(5)	176.1(2)	4.836(9)
9	DAWSIG	2.7686(13)	176.86(12)	4.855(3)
10	DIMXII	2.712(2)	170.49(16)	4.682(4)
11	DIMXOO	2.770(8)	176.5(3)	4.652(10)
12	EVIKOJ	3.264(7)	175.70(19)	5.33(1)

13	HOJWEL	2.7352(7)	174.60(5)	4.824(2)
14	HOJWIP	2.792(9)	177.4(3)	4.896(13)
		2.792(9)	176.5(3)	4.881(13)
		2.823(9)	174.4(3)	4.899(13)
		2.837(9)	173.2(3)	4.927(13)
15	HOJWOV	2.713(1)	174.89(7)	4.805(3)
16	HUVMES	2.7844(13)	174.45(5)	4.871(2)
17	LILMAW	2.741(2)	176.89(8)	4.833(3)
		2.808(2)	179.53(8)	4.895(3)
		2.807(4)	179.26(14)	4.898(6)
19	OCOMUO	2.754(2)	170.31(10)	4.822(3)
20	OCOMUO01	2.753(2)	170.23(10)	4.820(4)
21	OCOMUO02	2.7363(16)	169.94(8)	4.299(3)
22	OCOMUO03	2.7278(15)	169.89(7)	4.790(2)
23	OCOMUO04	2.7225(12)	169.80(6)	4.785(2)
24	OCOMUO05	2.7253(11)	169.85(4)	4.7916(14)
25	HISZIU	2.852(3)	173.78(12)	4.939(5)
26	JEFFUW	2.827(9)	175.1(4)	4.900(13)
27	QOXQAW	2.9400(12)	177.60(5)	5.0245(19)
28	ZEFKIG	2.817(4)	170.04(10)	4.890(6)
		2.814(4)	170.39(12)	4.882(6)
29	ZEFKOM	2.801(3)	176.73(13)	4.891(5)
		2.814(3)	169.59(10)	4.889(4)

Table S2: Halogen bond parameters between perfluorohaloalkanes and aromatic/aliphatic N-oxides.

S.No	**CSD-Refcode**	***d*(I···O) [Å]**	**∠(C–I···O) [°]**	***d*(C···O) [Å]**
1	LAZZAQ	2.944(11)	173.2(4)	5.090(14)
		2.769(12)	172.6(4)	4.917(15)
		2.980(15)	170.9(5)	5.125(16)
		2.856(11)	173.1(8)	5.007(18)
		2.875(16)	177.9(8)	5.035(19)
		2.777(15)	171.6(8)	4.933(19)
		2.856((11)	17.1(8)	5.007(18)
		2.892(12)	178.4(4)	5.053(15)
		2.775(13)	176.3(5)	4.937(16)
		2.875(11)	170.2(5)	5.008(15)
2	LAZZIY	2.879(4)	179.32(18)	5.033(7)
		2.804(6)	175.1(3)	4.962(10)
		2.853(7)	173.4(3)	5.01(1)
		2.862(6)	170.5(3)	5.00(1)
		2.816(7)	168.9(4)	4.947(10)
		2.833(7)	172.7(3)	4.996(9)
3	LAZYOD	2.744(2)	173.63(10)	4.889(4)
		2.810(3)	170.57(9)	4.948(5)
		2.874(3)	172.95(15)	5.015(4)
		2.836(2)	170.90(11)	4.976(4)
		2.860(11)	172.4(6)	5.003(10)
		2.786(3)	178.14(10)	4.944(5)
		2.703(2)	175.39(12)	4.854(4)
		2.805(2)	178.68(9)	4.963(4)
		2.825(3)	176.12(10)	4.974(5)
4	LAZYUJ	2.788(6)	173.2(3)	4.927(11)

		2.808(5)	172.1(3)	4.953(11)
		2.831(5)	173.0(3)	4.98(1)
5	LULQOA	2.8148(17)	171.18(8)	4.948(4)

5. Computational Data

Full *Gaussian 16* reference:

Gaussian 16, Revision C.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2019.

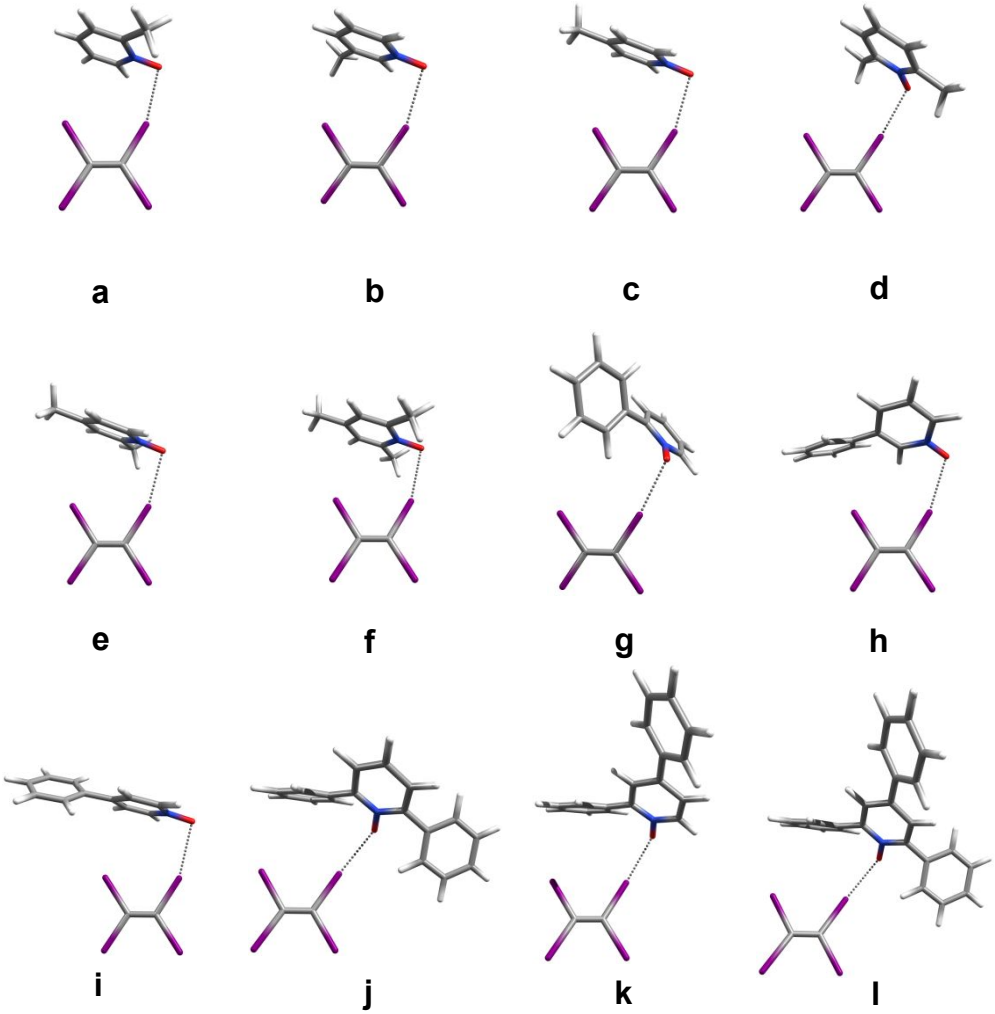

Figure S6. Optimized C_2I_4 XB complex structures with different pyridine-N-oxide XB acceptors a) **2**, b) **3**, c) **4**, d) **5**, e) **6**, f) **7**, g) **8**, h) **9**, i) **10**, j) **11**, k) **12**, l) **13**.

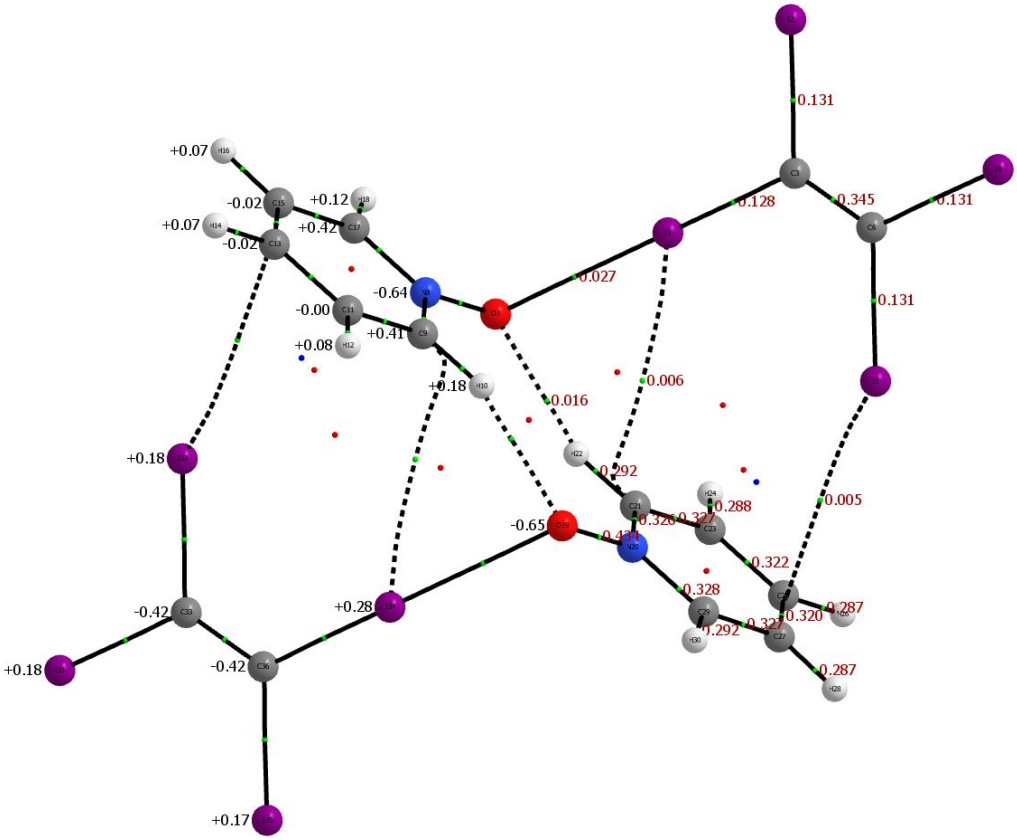

Figure S7. QTAIM bond paths, bond critical points (green) and their electron densities (red text) and atomic charges (black text) in PBE0-D3/def2-TZVP optimized structure of two C_2I_4 and two **1** molecules showing the halogen bonds and other weaker intermolecular interactions.

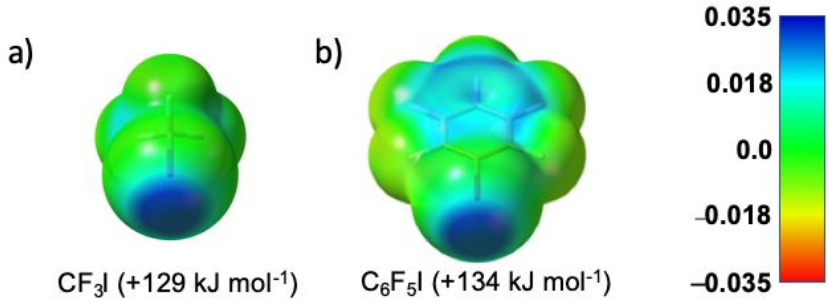

Figure S8. Computed electrostatic potentials projected on the 0.001 a.u. electron density surfaces of donors (a) CF_3I, and (b) C_6F_5I with $V_{S,max}$ values.

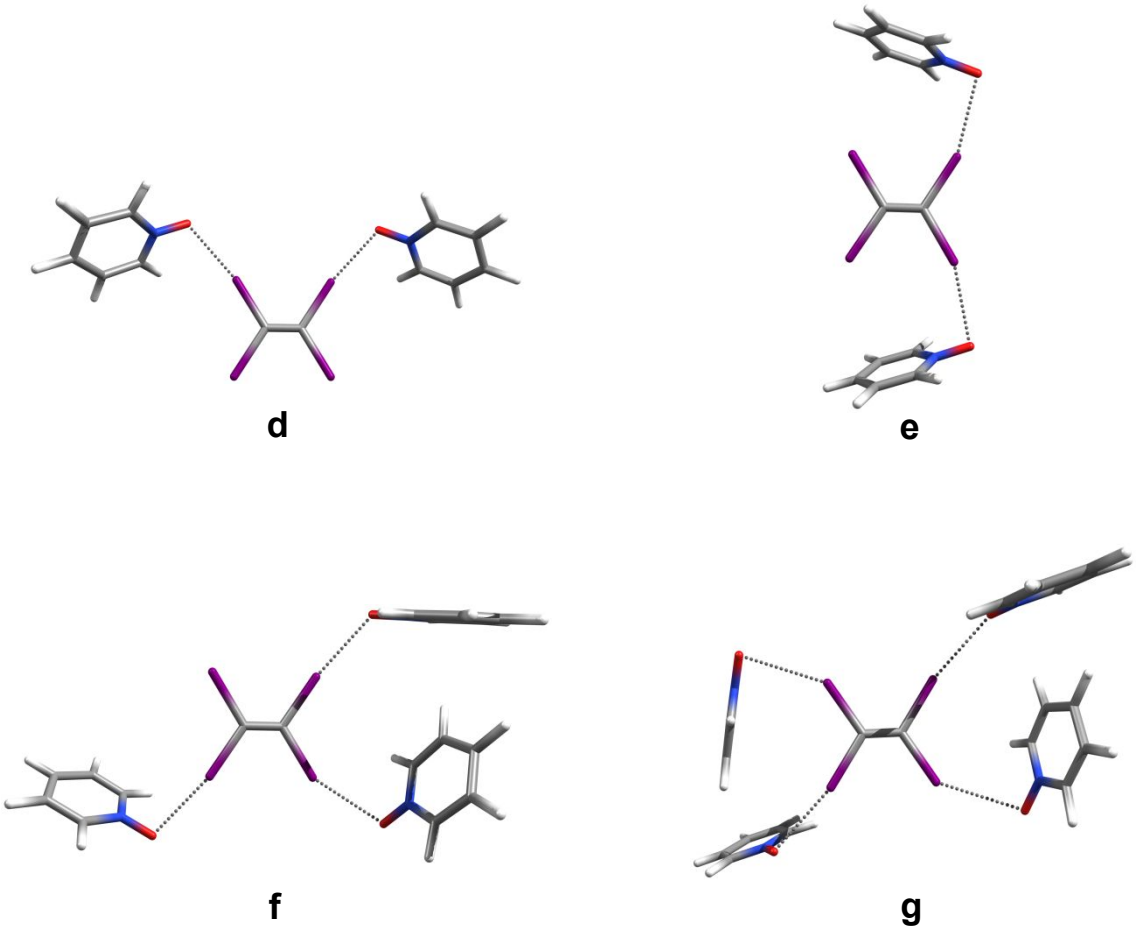

Figure S9. Different XB motifs of **1** and C_2I_4 optimized at PBE0-D3/def2-TZVP level of theory.

Table S3. Distances and XB motif energies for **1**·C_2I_4.

Motif	I···O [Å]	ΔE_{int} [kJ mol^{-1}]
a	2.756	31.9
b	2.837 – 2.913	28.9
c	2.935 – 2.981	26.5
d	2.803	29.2
e	2.901	32.4
f	2.826 - 3.015	28.7
g	2.953 – 3.212	29.9

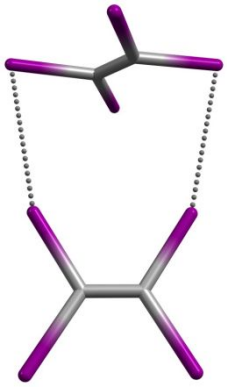

Figure S10. C–I···I′–C XB bonded structure of two C_2I_4 optimized at PBE0-D3/def2-TZVP level of theory (d(I···I) = 3.770 Å, and ΔE_{int} = 15.2 kJ mol^{-1}).

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