Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Crystal Data

For

Synthesis and Energetic Properties of Homocubane Based High Energy Density Materials

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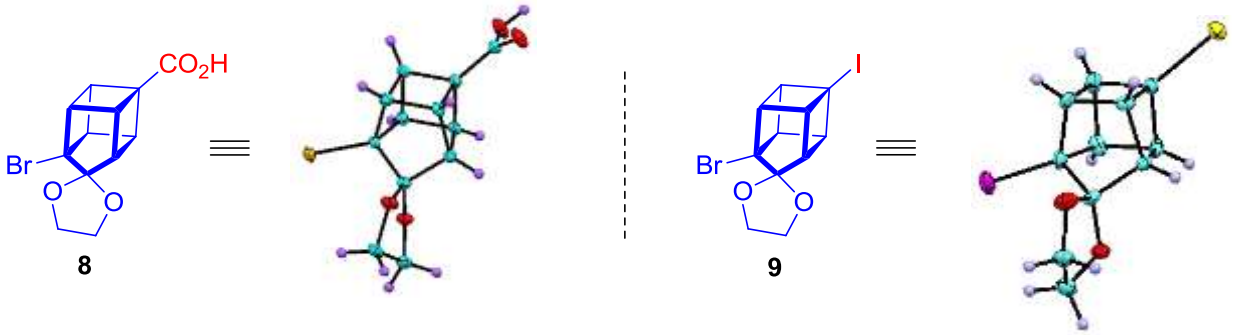
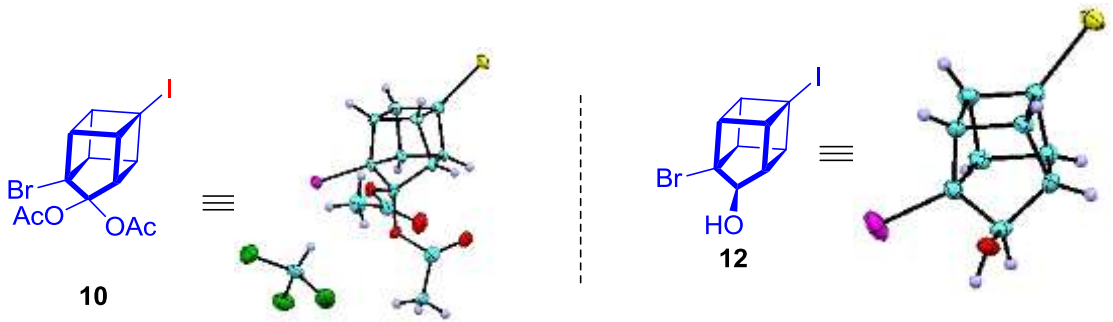
Table XR1. Single crystal X-ray data and structure refinement for compounds 8 and 9.



Identification code	**8**	**9**
CCDC	1967802	1967801
Empirical formula	$C_{12}H_{11}BrO_4$	$C_{11}H_{10}BrIO_2$
Formula weight	299.12	381.00
Temperature/K	150	150
Crystal system	triclinic	monoclinic
Space group	P-1	$P2_1/c$
a/Å	5.8311(2)	14.4803
b/Å	7.7293(3)	6.2437
c/Å	12.1411(5)	12.7594
α/°	100.726(3)	90
β/°	100.614(3)	106.322
γ/°	90.063(3)	90
Volume/Å^3	528.09(4)	1107.09
Z	2	4
ρ_{calc}g/cm^3	1.881	2.286

μ/mm^{-1}	3.891	6.477
F(000)	300.0	720.0
Crystal size/mm^3	0.203 × 0.132 × 0.09	0.168 × 0.181 × 0.111
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.816 to 62.554	5.864 to 49.988
Index ranges	$-8 \le h \le 8$, $-11 \le k \le 11$, $-17 \le l \le 17$	$-17 \le h \le 17$, $-7 \le k \le 7$, $-15 \le l \le 15$
Reflections collected	14118	11920
Independent reflections	3159 [R_{int} = 0.0386, R_{sigma} = 0.0254]	1902 [R_{int} = 0.0303, R_{sigma} = 0.0164]
Data/restraints/parameters	3159/0/158	1899/0/136
Goodness-of-fit on F^2	1.063	1.073
Final R indexes [I>=2σ (I)]	R_1 = 0.0259, wR_2 = 0.0590	R_1 = 0.0182, wR_2 = 0.0441
Final R indexes [all data]	R_1 = 0.0292, wR_2 = 0.0606	R_1 = 0.0195, wR_2 = 0.0446
Largest diff. peak/hole / e $Å^{-3}$	0.43/-0.49	0.44/-0.53

Table XR2. Single crystal X-ray data and structure refinement for compounds 10 and 12.

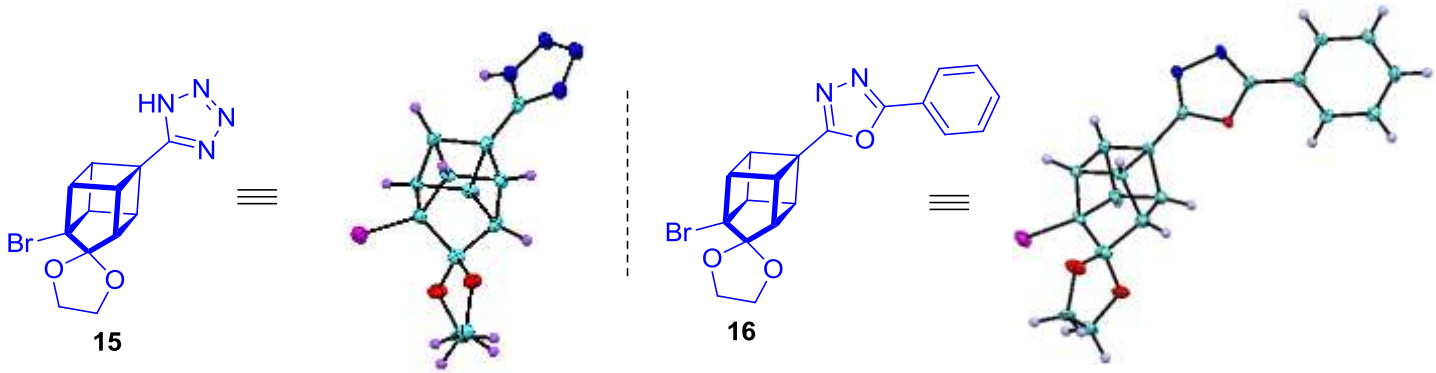


Identification code	**10**	**12**

CCDC	1964255	1964254
Empirical formula	$C_{14}H_{13}BrCl_3IO_4$	C_9H_8BrIO
Formula weight	558.40	338.96
Temperature/K	150	150
Crystal system	monoclinic	monoclinic
Space group	$P2_1/n$	$P2_1/c$
a/Å	9.9006(5)	6.6943(5)
b/Å	9.5204(5)	22.5033(16)
c/Å	19.1866(11)	19.5658(13)
α/°	90	90
β/°	93.750(5)	98.063(7)
γ/°	90	90
Volume/Å^3	1804.61(17)	2918.3(4)
Z	4	4
ρ_{calc}g/cm^3	2.055	2.314
μ/mm^{-1}	4.447	7.348
F(000)	1072.0	1896.0
Crystal size/mm^3	0.171 × 0.140 × 0.037	0.315 × 0.139 × 0.063
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.762 to 49.996	5.55 to 50
Index ranges	$-11 \le h \le 11$, $-11 \le k \le 11$, $-22 \le l \le 22$	$-7 \le h \le 5$, $-26 \le k \le 24$, $-22 \le l \le 23$
Reflections collected	18421	10139
Independent reflections	3178 [R_{int} = 0.0973, R_{sigma} = 0.0755]	5017 [R_{int} = 0.0265, R_{sigma} = 0.0307]
Data/restraints/parameters	3178/0/210	5017/0/331
Goodness-of-fit on F^2	1.044	1.035

Final R indexes [I>=2σ (I)]	R_1 = 0.0454, wR_2= 0.0640	R_1 = 0.0284, wR_2 = 0.0622
Final R indexes [all data]	R_1 = 0.0756, wR_2 = 0.0732	R_1 = 0.0343, wR_2 = 0.0654
Largest diff. peak/hole / e Å^{-3}	0.76/-0.64	1.23/-0.88

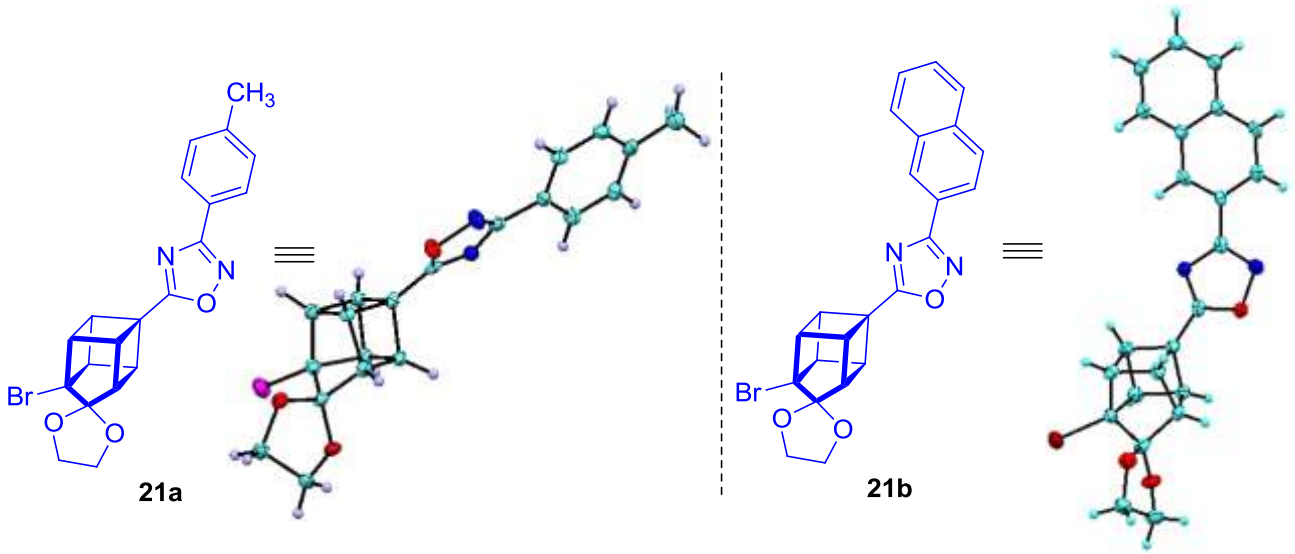
Table XR3. Single crystal X-ray data and structure refinement for compounds 12 and 16.



Identification code	**15**	**16**
CCDC	1964252	1964251
Empirical formula	$C_{12}H_{11}BrN_4O_2$	$C_{19}H_{15}BrN_2O_3$
Formula weight	323.16	399.24
Temperature/K	150	150
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/n$
a/Å	16.0595(9)	7.2387(3)
b/Å	9.6446(6)	11.2396(4)
c/Å	7.9955(4)	38.4923(15)
α/°	90	90
β/°	96.110(5)	91.568(4)

γ/°	90	90
Volume/Å^3	1231.37(12)	3130.6(2)
Z	4	8
ρ_{calc}g/cm^3	1.743	1.694
μ/mm^{-1}	3.342	2.649
F(000)	648.0	1616.0
Crystal size/mm^3	0.209 × 0.185 × 0.026	0.201 × 0.065 × 0.039
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.934 to 49.992	3.776 to 49.998
Index ranges	$-19 \leq h \leq 14$, $-11 \leq k \leq 11$, $-9 \leq l \leq 9$	$-8 \leq h \leq 6$, $-13 \leq k \leq 13$, $-45 \leq l \leq 45$
Reflections collected	5984	27220
Independent reflections	2167 [R_{int} = 0.0549, R_{sigma} = 0.0615]	5503 [R_{int} = 0.0824, R_{sigma} = 0.0644]
Data/restraints/parameters	2167/0/172	5503/0/451
Goodness-of-fit on F^2	1.041	1.096
Final R indexes [I>=2σ (I)]	R_1 = 0.0377, wR_2 = 0.0900	R_1 = 0.0514, wR_2 = 0.1085
Final R indexes [all data]	R_1 = 0.0518, wR_2 = 0.0976	R_1 = 0.0690, wR_2 = 0.1180
Largest diff. peak/hole / e Å^{-3}	0.45/-0.60	1.10/-0.58

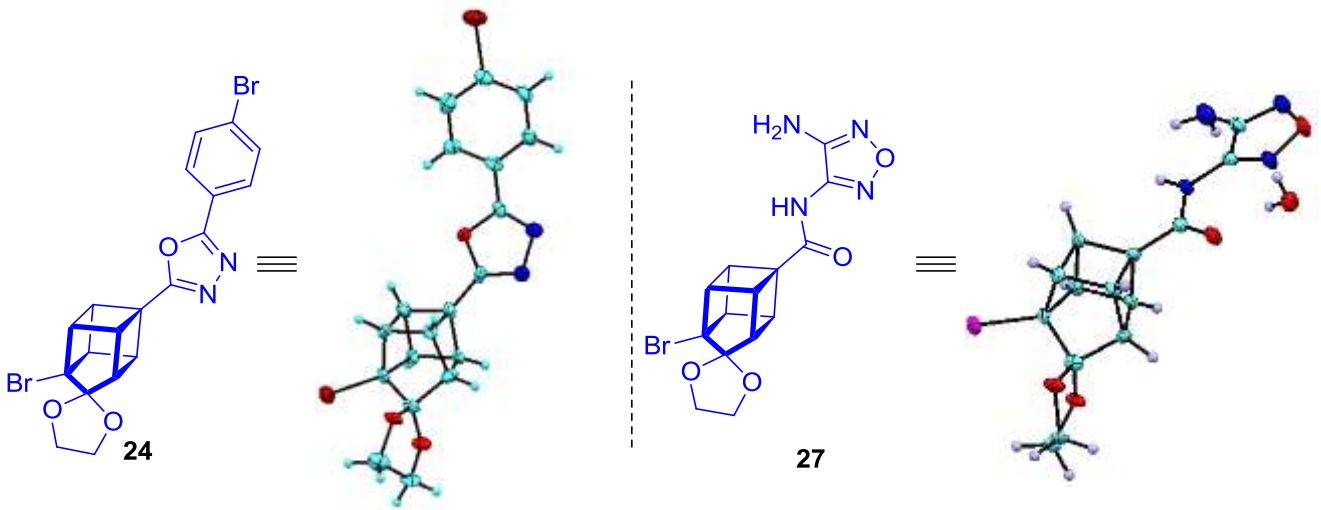
Table XR4. Single crystal X-ray data and structure refinement for compounds 21a and 21b.



Identification code	**21a**	**21b**
CCDC	1964250	1982149
Empirical formula	$C_{20}H_{17}BrN_2O_3$	$C_{23}H_{17}BrN_2O_3$
Formula weight	413.26	449.29
Temperature/K	150	150.0
Crystal system	triclinic	monoclinic
Space group	P-1	$P2_1/n$
a/Å	6.3380(3)	6.1192(3)
b/Å	8.3852(4)	25.3018(12)
c/Å	16.4495(8)	12.0093(6)
α/°	83.445(4)	90

β/°	82.649(4)	92.571(5)
γ/°	79.039(4)	90
Volume/Å^3	847.58(8)	1857.49(16)
Z	2	4
ρ_{calc}g/cm^3	1.619	1.607
μ/mm^{-1}	2.449	2.242
F(000)	420.0	912.0
Crystal size/mm^3	0.331 × 0.063 × 0.035	0.208 × 0.125 × 0.103
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.97 to 50	4.68 to 49.988
Index ranges	$-7 \le h \le 5$, $-9 \le k \le 9$, $-19 \le l \le 19$	$-6 \le h \le 7$, $-30 \le k \le 28$, $-14 \le l \le 14$
Reflections collected	5492	10517
Independent reflections	2929 [R_{int} = 0.0498, R_{sigma} = 0.0558]	3271 [R_{int} = 0.0505, R_{sigma} = 0.0492]
Data/restraints/parameters	2929/0/236	3271/0/262
Goodness-of-fit on F^2	1.041	1.056
Final R indexes [I>=2σ (I)]	R_1 = 0.0376, wR_2 = 0.0806	R_1 = 0.0363, wR_2 = 0.0768
Final R indexes [all data]	R_1 = 0.0463, wR_2 = 0.0858	R_1 = 0.0526, wR_2 = 0.0843
Largest diff. peak/hole / e Å^{-3}	0.36/-0.50	0.37/-0.43

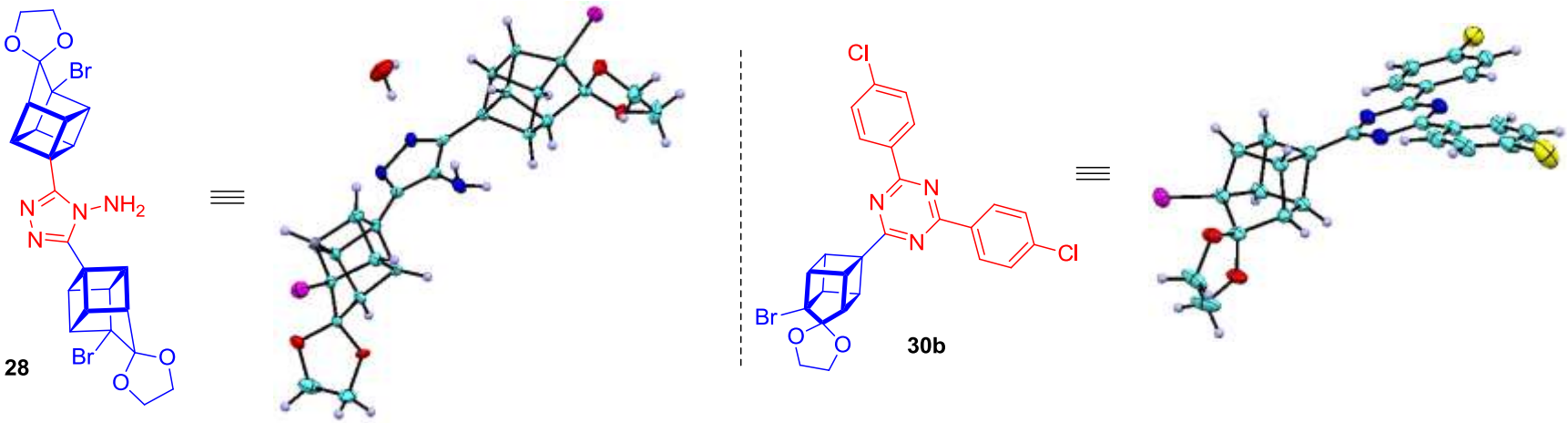
Table XR5. Single crystal X-ray data and structure refinement for compounds 24 and 27.



Identification code	**24**	**27**
CCDC	1982155	1963901
Empirical formula	$C_{19}H_{14}Br_2N_2O_3$	$C_{14}H_{13}BrN_4O_4.H_2O$
Formula weight	478.14	399.21
Temperature/K	150.0	150
Crystal system	monoclinic	triclinic
Space group	$P2_1/c$	P-1
a/Å	22.348(2)	5.8063(3)
b/Å	6.2154(6)	6.9352(4)
c/Å	12.4894(11)	19.2746(11)
α/°	90	86.817(4)
β/°	90.245(9)	85.678(5)

γ/°	90	72.799(5)
Volume/Å^3	1734.8(3)	738.87(7)
Z	4	2
ρ_{calc}g/cm^3	1.831	1.794
μ/mm^{-1}	4.695	2.818
F(000)	944.0	404.0
Crystal size/mm^3	0.179 × 0.126 × 0.092	0.2 × 0.11 × 0.05
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.524 to 49.994	6.154 to 50
Index ranges	-26 ≤ h ≤ 26, -7 ≤ k ≤ 7, -14 ≤ l ≤ 14	-6 ≤ h ≤ 6, -8 ≤ k ≤ 8, -14 ≤ l ≤ 22
Reflections collected	18506	5842
Independent reflections	3069 [R_{int} = 0.1694, R_{sigma} = 0.1277]	2589 [Rint = 0.0734, Rsigma = 0.0782]
Data/restraints/parameters	3069/0/235	2589/0/221
Goodness-of-fit on F^2	1.050	1.073
Final R indexes [I>=2σ (I)]	R_1 = 0.0698, wR_2 = 0.1310	R1 = 0.0454, wR2 = 0.0848
Final R indexes [all data]	R_1 = 0.1246, wR_2 = 0.1684	R1 = 0.0601, wR2 = 0.0928
Largest diff. peak/hole / e Å^{-3}	0.97/-0.97	0.78/-0.60

Table XR6. Single crystal X-ray data and structure refinement for compounds 28 and 30b.



Identification code	**28**	**30b**
CCDC	1973818	1963891
Empirical formula	$C_{24}H_{24}Br_2N_4O_5$	$C_{26}H_{18}BrCl_2N_3O_2$
Formula weight	608.29	555.24
Temperature/K	150.0	150 K
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	P21/n
a/Å	10.5956(5)	5.90980(10)
b/Å	22.2440(10)	23.7889(6)
c/Å	10.0485(4)	16.2608(4)
α/°	90	90
β/°	107.185(5)	96.123(2)
γ/°	90	90

Volume/Å^3	2262.58(18)	2273.03(9)
Z	4	4
ρ_{calc}g/cm^3	1.7856	1.623
μ/mm^{-1}	3.629	2.075
F(000)	1222.3	1120.0
Crystal size/mm^3	0.157 × 0.102 × 0.059	0.21 × 0.141 × 0.089
Radiation	Mo Kα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.66 to 50	5.322 to 50
Index ranges	-14 ≤ h ≤ 14, -32 ≤ k ≤ 30, -14 ≤ l ≤ 13	-7 ≤ h ≤ 7, -28 ≤ k ≤ 28, -19 ≤ l ≤ 19
Reflections collected	24988	21939
Independent reflections	3969 [R_{int} = 0.0869, R_{sigma} = 0.1068]	3917 [Rint = 0.0277, Rsigma = 0.0169]
Data/restraints/parameters	3969/0/320	3917/0/307
Goodness-of-fit on F^2	1.066	1.033
Final R indexes [I>=2σ (I)]	R_1 = 0.0522, wR_2 = 0.1148	R1 = 0.0258, wR2 = 0.0644
Final R indexes [all data]	R_1 = 0.0732, wR_2 = 0.1259	R1 = 0.0285, wR2 = 0.0659
Largest diff. peak/hole / e Å^{-3}	1.06/-0.85	0.34/-0.30