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Supporting information for article:

Trioxazolo[23]metacyclophane: synthesis, structural analysis, and optical properties

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Supporting Information for

Trioxazolo[2^3]metacyclophane: Synthesis, Structural Analysis, and Optical Properties

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Contents:

1. ¹H and ¹³C Spectra

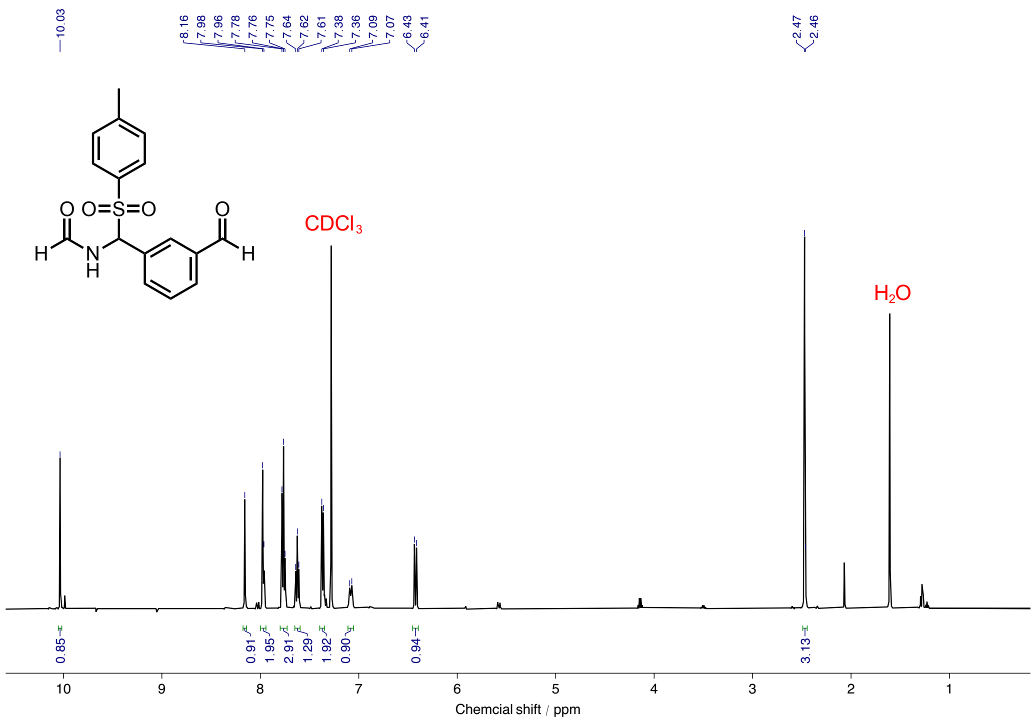

Figure S1. ¹H NMR spectrum of **1** (500 MHz, CDCl$_3$).

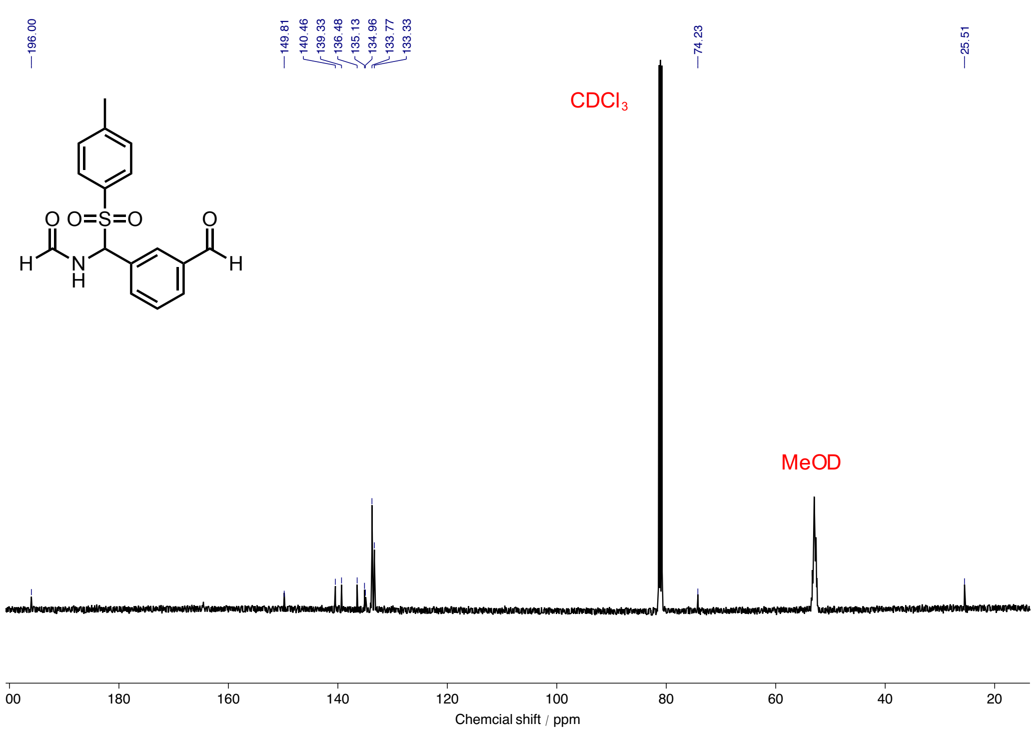

Figure S2. ¹³C NMR spectrum of **1** (125 MHz, 9:1 CDCl$_3$/MeOD).

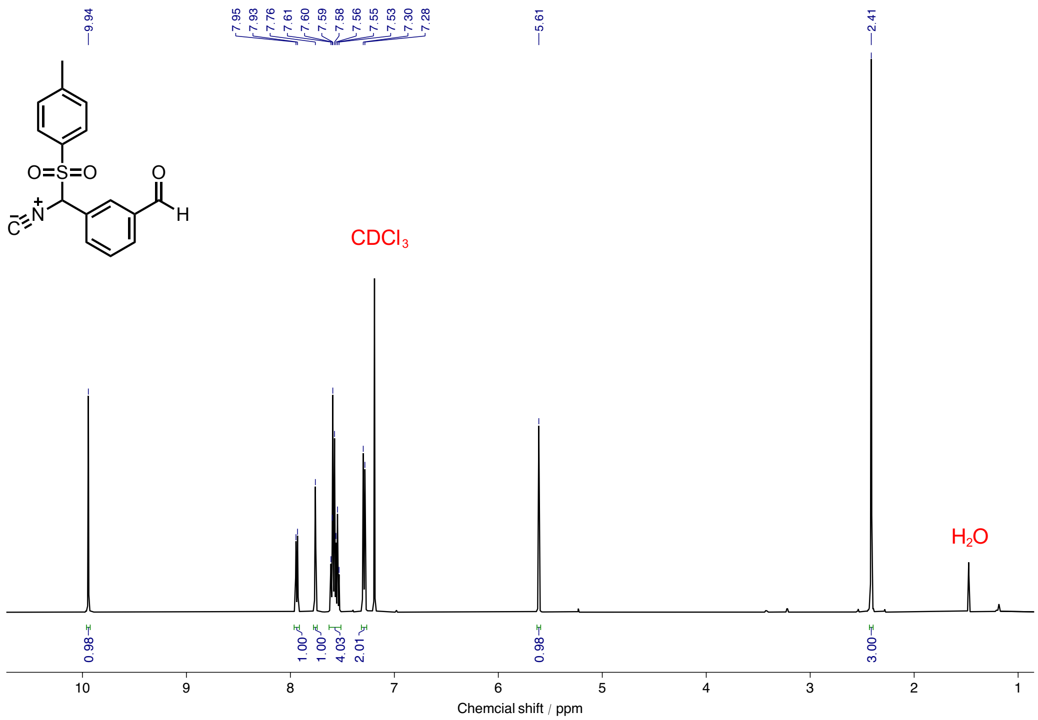

Figure S3. ^{1}H NMR spectrum of **2** (500 MHz, $CDCl_3$).

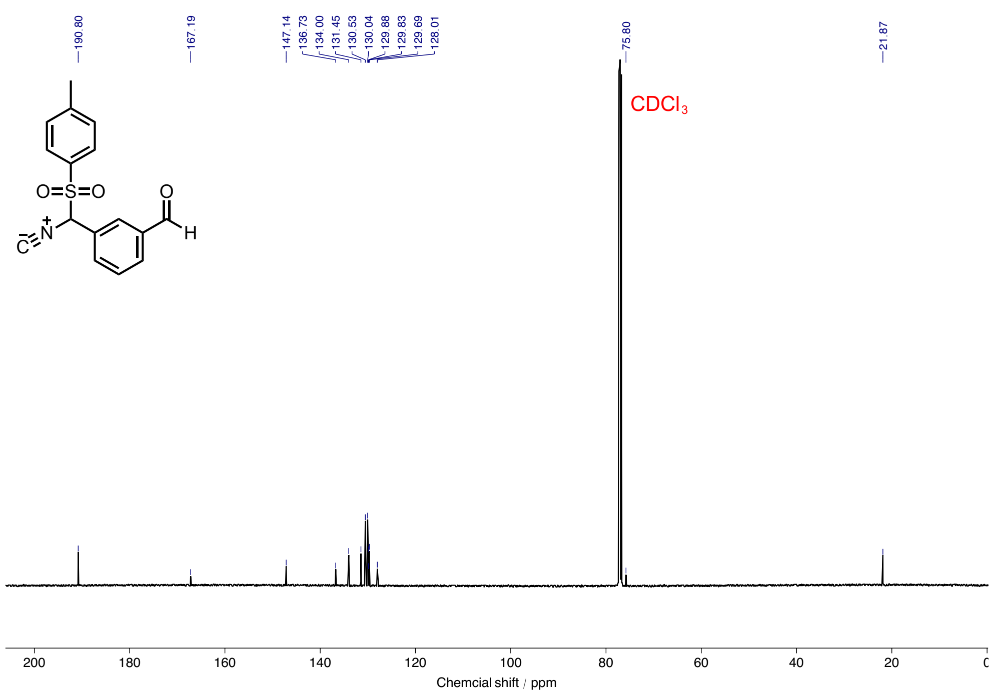

Figure S4. ^{13}C NMR spectrum of **2** (125 MHz, $CDCl_3$).

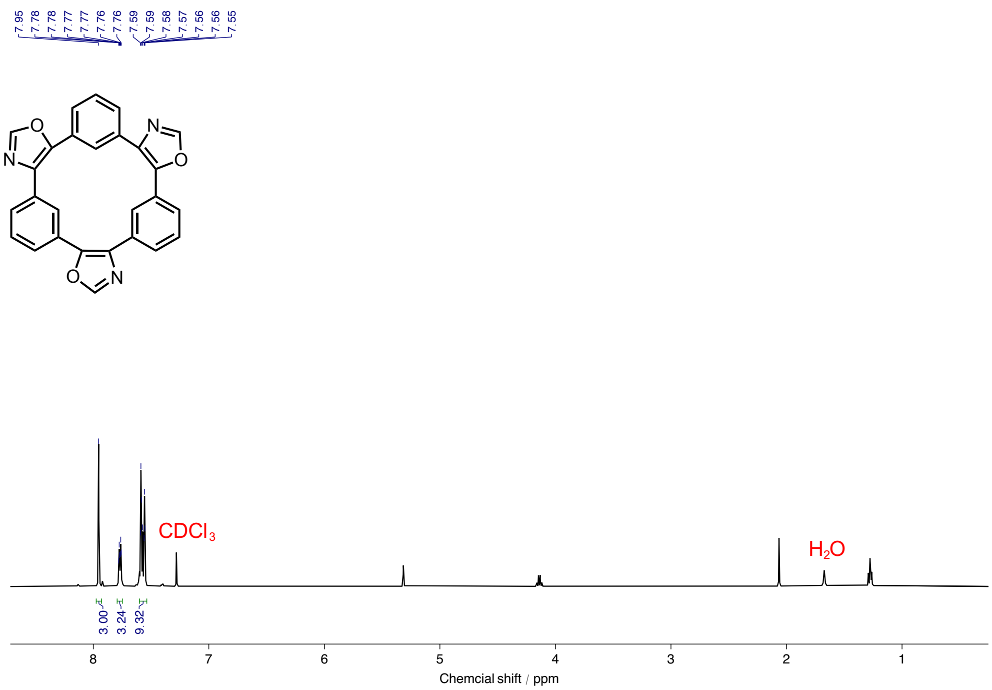

Figure S5. ^{1}H NMR spectrum of **M** (500 MHz, $CDCl_3$).

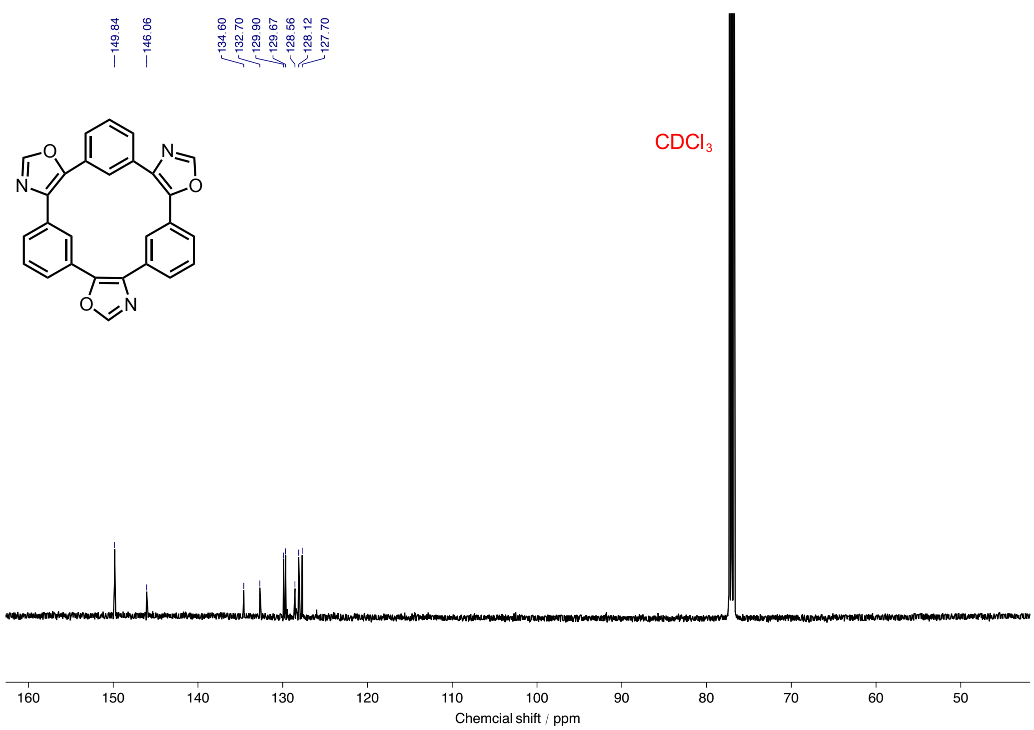

Figure S6. ^{13}C NMR spectrum of **M** (125 MHz, $CDCl_3$).

2. Mass Spectra

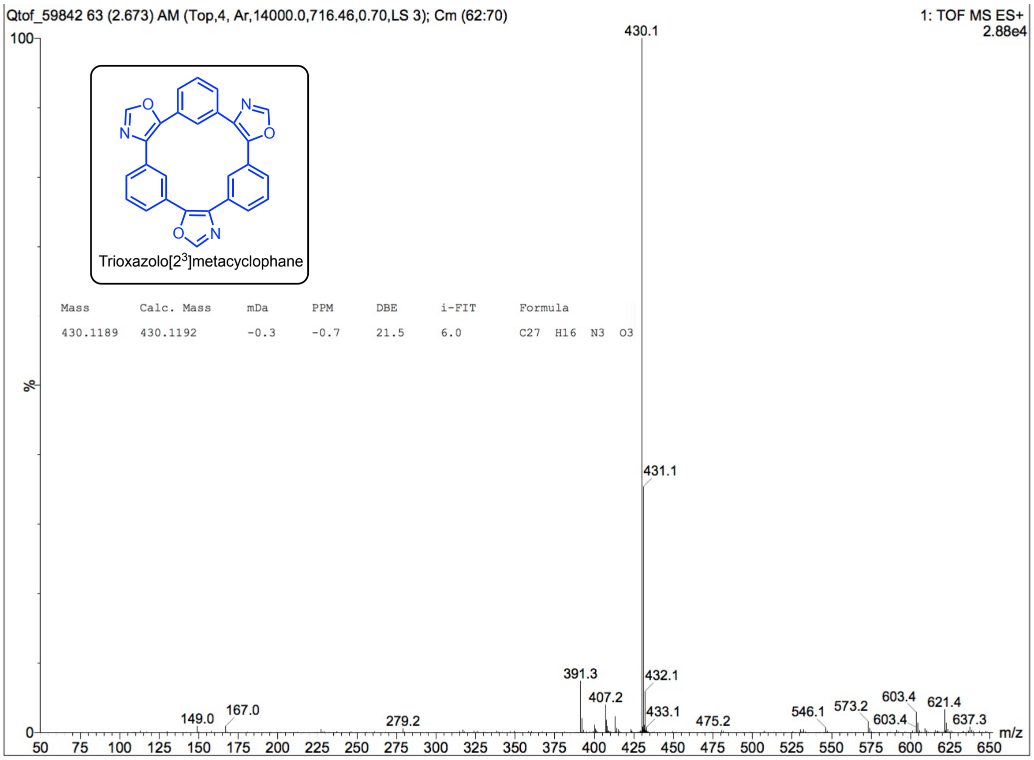

Figure S7. Mass spectrum of **M**.

3. Additional Crystallographic Data

Table S1. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for **M**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2552(1)	11796(2)	5566(1)	31(1)
O(2)	5206(1)	9142(2)	9081(1)	23(1)
O(3)	1447(1)	2283(2)	7509(1)	25(1)
N(1)	1069(1)	10843(2)	5606(1)	20(1)
N(2)	5951(1)	9435(2)	8022(1)	16(1)
N(3)	2160(1)	2795(2)	8574(1)	18(1)
C(1)	1649(1)	12051(2)	5376(1)	29(1)
C(2)	2544(1)	10216(2)	5969(1)	20(1)
C(3)	3476(1)	9537(2)	6197(1)	18(1)
C(4)	4110(1)	8996(2)	5658(1)	20(1)

C(5)	5009(1)	8421(2)	5848(1)	21(1)
C(6)	5292(1)	8398(2)	6572(1)	18(1)
C(7)	4672(1)	8951(2)	7119(1)	16(1)
C(8)	3766(1)	9524(2)	6926(1)	17(1)
C(9)	5005(1)	9019(2)	7880(1)	16(1)
C(10)	6012(1)	9496(2)	8728(1)	21(1)
C(11)	4559(1)	8824(2)	8528(1)	17(1)
C(12)	3613(1)	8241(2)	8742(1)	16(1)
C(13)	3152(1)	8982(2)	9342(1)	18(1)
C(14)	2285(1)	8292(2)	9554(1)	21(1)
C(15)	1876(1)	6858(2)	9186(1)	20(1)
C(16)	2334(1)	6107(2)	8586(1)	16(1)
C(17)	3193(1)	6826(2)	8363(1)	16(1)
C(18)	1992(1)	4443(2)	8228(1)	18(1)
C(19)	1824(1)	1612(2)	8121(1)	22(1)
C(20)	1564(1)	4127(2)	7579(1)	18(1)
C(21)	1191(1)	5233(2)	6984(1)	18(1)
C(22)	421(1)	4601(2)	6582(1)	22(1)
C(23)	44(1)	5636(2)	6024(1)	23(1)
C(24)	419(1)	7301(2)	5860(1)	21(1)
C(25)	1210(1)	7937(2)	6243(1)	18(1)
C(26)	1589(1)	6899(2)	6806(1)	18(1)
C(27)	1637(1)	9627(2)	5993(1)	19(1)

Table S2. Bond lengths [Å] and angles [°] for **M**.

O(1)-C(1)	1.339(2)
O(1)-C(2)	1.389(2)
O(2)-C(10)	1.343(2)
O(2)-C(11)	1.3852(19)
O(3)-C(19)	1.337(2)
O(3)-C(20)	1.389(2)
N(1)-C(1)	1.291(2)
N(1)-C(27)	1.401(2)
N(2)-C(10)	1.297(2)
N(2)-C(9)	1.400(2)
N(3)-C(19)	1.299(2)
N(3)-C(18)	1.400(2)
C(1)-H(1)	0.9500
C(2)-C(27)	1.361(2)
C(2)-C(3)	1.474(2)
C(3)-C(8)	1.396(2)
C(3)-C(4)	1.399(2)
C(4)-C(5)	1.388(2)
C(4)-H(4)	0.9500
C(5)-C(6)	1.385(2)
C(5)-H(5)	0.9500
C(6)-C(7)	1.400(2)
C(6)-H(6)	0.9500
C(7)-C(8)	1.398(2)
C(7)-C(9)	1.471(2)
C(8)-H(8)	0.9500
C(9)-C(11)	1.357(2)
C(10)-H(10)	0.9500
C(11)-C(12)	1.466(2)
C(12)-C(17)	1.394(2)
C(12)-C(13)	1.398(2)
C(13)-C(14)	1.389(2)
C(13)-H(13)	0.9500
C(14)-C(15)	1.388(2)
C(14)-H(14)	0.9500

C(15)-C(16)	1.398(2)
C(15)-H(15)	0.9500
C(16)-C(17)	1.394(2)
C(16)-C(18)	1.482(2)
C(17)-H(17)	0.9500
C(18)-C(20)	1.354(2)
C(19)-H(19)	0.9500
C(20)-C(21)	1.464(2)
C(21)-C(22)	1.395(2)
C(21)-C(26)	1.401(2)
C(22)-C(23)	1.385(2)
C(22)-H(22)	0.9500
C(23)-C(24)	1.384(3)
C(23)-H(23)	0.9500
C(24)-C(25)	1.403(2)
C(24)-H(24)	0.9500
C(25)-C(26)	1.395(2)
C(25)-C(27)	1.471(2)
C(26)-H(26)	0.9500
C(1)-O(1)-C(2)	104.33(14)
C(10)-O(2)-C(11)	104.06(12)
C(19)-O(3)-C(20)	104.20(13)
C(1)-N(1)-C(27)	104.48(14)
C(10)-N(2)-C(9)	104.56(13)
C(19)-N(3)-C(18)	104.17(13)
N(1)-C(1)-O(1)	115.25(15)
N(1)-C(1)-H(1)	122.4
O(1)-C(1)-H(1)	122.4
C(27)-C(2)-O(1)	107.46(14)
C(27)-C(2)-C(3)	136.72(15)
O(1)-C(2)-C(3)	115.54(14)
C(8)-C(3)-C(4)	119.20(15)
C(8)-C(3)-C(2)	122.14(15)
C(4)-C(3)-C(2)	118.53(14)
C(5)-C(4)-C(3)	120.32(15)
C(5)-C(4)-H(4)	119.8

C(3)-C(4)-H(4)	119.8
C(6)-C(5)-C(4)	120.35(15)
C(6)-C(5)-H(5)	119.8
C(4)-C(5)-H(5)	119.8
C(5)-C(6)-C(7)	120.22(15)
C(5)-C(6)-H(6)	119.9
C(7)-C(6)-H(6)	119.9
C(8)-C(7)-C(6)	119.27(14)
C(8)-C(7)-C(9)	121.27(14)
C(6)-C(7)-C(9)	119.36(14)
C(3)-C(8)-C(7)	120.63(15)
C(3)-C(8)-H(8)	119.7
C(7)-C(8)-H(8)	119.7
C(11)-C(9)-N(2)	108.24(14)
C(11)-C(9)-C(7)	132.62(15)
N(2)-C(9)-C(7)	119.08(14)
N(2)-C(10)-O(2)	114.99(14)
N(2)-C(10)-H(10)	122.5
O(2)-C(10)-H(10)	122.5
C(9)-C(11)-O(2)	108.14(14)
C(9)-C(11)-C(12)	134.39(15)
O(2)-C(11)-C(12)	117.26(13)
C(17)-C(12)-C(13)	119.35(15)
C(17)-C(12)-C(11)	118.62(14)
C(13)-C(12)-C(11)	121.89(15)
C(14)-C(13)-C(12)	119.54(16)
C(14)-C(13)-H(13)	120.2
C(12)-C(13)-H(13)	120.2
C(15)-C(14)-C(13)	121.13(15)
C(15)-C(14)-H(14)	119.4
C(13)-C(14)-H(14)	119.4
C(14)-C(15)-C(16)	119.65(15)
C(14)-C(15)-H(15)	120.2
C(16)-C(15)-H(15)	120.2
C(17)-C(16)-C(15)	119.27(15)
C(17)-C(16)-C(18)	118.39(14)
C(15)-C(16)-C(18)	121.98(14)

C(12)-C(17)-C(16)	121.02(15)
C(12)-C(17)-H(17)	119.5
C(16)-C(17)-H(17)	119.5
C(20)-C(18)-N(3)	108.64(14)
C(20)-C(18)-C(16)	132.68(15)
N(3)-C(18)-C(16)	118.58(13)
N(3)-C(19)-O(3)	115.20(15)
N(3)-C(19)-H(19)	122.4
O(3)-C(19)-H(19)	122.4
C(18)-C(20)-O(3)	107.79(14)
C(18)-C(20)-C(21)	135.76(16)
O(3)-C(20)-C(21)	116.41(14)
C(22)-C(21)-C(26)	119.41(15)
C(22)-C(21)-C(20)	118.82(15)
C(26)-C(21)-C(20)	121.77(15)
C(23)-C(22)-C(21)	120.00(16)
C(23)-C(22)-H(22)	120.0
C(21)-C(22)-H(22)	120.0
C(24)-C(23)-C(22)	120.79(16)
C(24)-C(23)-H(23)	119.6
C(22)-C(23)-H(23)	119.6
C(23)-C(24)-C(25)	120.02(15)
C(23)-C(24)-H(24)	120.0
C(25)-C(24)-H(24)	120.0
C(26)-C(25)-C(24)	119.20(15)
C(26)-C(25)-C(27)	123.21(15)
C(24)-C(25)-C(27)	117.46(14)
C(25)-C(26)-C(21)	120.53(15)
C(25)-C(26)-H(26)	119.7
C(21)-C(26)-H(26)	119.7
C(2)-C(27)-N(1)	108.48(15)
C(2)-C(27)-C(25)	132.57(15)
N(1)-C(27)-C(25)	118.31(14)

Table S3. Anisotropic displacement parameters ($Å^2$x 10^3) for **M**. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	37(1)	24(1)	31(1)	8(1)	-13(1)	-7(1)
O(2)	24(1)	25(1)	19(1)	1(1)	-3(1)	-1(1)
O(3)	27(1)	20(1)	27(1)	2(1)	2(1)	-2(1)
N(1)	28(1)	15(1)	18(1)	2(1)	-8(1)	2(1)
N(2)	14(1)	16(1)	16(1)	3(1)	-2(1)	0(1)
N(3)	17(1)	17(1)	19(1)	5(1)	2(1)	-1(1)
C(1)	37(1)	20(1)	29(1)	5(1)	-18(1)	0(1)
C(2)	29(1)	16(1)	14(1)	1(1)	-5(1)	-2(1)
C(3)	22(1)	12(1)	18(1)	2(1)	-2(1)	-5(1)
C(4)	31(1)	17(1)	13(1)	0(1)	-1(1)	-7(1)
C(5)	27(1)	18(1)	19(1)	-3(1)	7(1)	-6(1)
C(6)	19(1)	14(1)	23(1)	0(1)	3(1)	-3(1)
C(7)	20(1)	13(1)	16(1)	1(1)	0(1)	-3(1)
C(8)	20(1)	15(1)	15(1)	1(1)	2(1)	-2(1)
C(9)	16(1)	13(1)	19(1)	1(1)	-2(1)	1(1)
C(10)	18(1)	21(1)	23(1)	3(1)	-3(1)	-1(1)
C(11)	20(1)	14(1)	17(1)	-1(1)	-4(1)	1(1)
C(12)	19(1)	16(1)	13(1)	5(1)	-1(1)	4(1)
C(13)	25(1)	17(1)	13(1)	2(1)	-1(1)	4(1)
C(14)	27(1)	19(1)	15(1)	2(1)	6(1)	8(1)
C(15)	19(1)	20(1)	19(1)	7(1)	4(1)	3(1)
C(16)	19(1)	15(1)	15(1)	5(1)	-1(1)	3(1)
C(17)	19(1)	17(1)	12(1)	2(1)	1(1)	4(1)
C(18)	15(1)	19(1)	20(1)	4(1)	5(1)	1(1)
C(19)	22(1)	18(1)	26(1)	6(1)	4(1)	1(1)
C(20)	17(1)	17(1)	22(1)	2(1)	4(1)	-1(1)
C(21)	19(1)	20(1)	16(1)	-1(1)	2(1)	3(1)
C(22)	21(1)	23(1)	23(1)	0(1)	2(1)	-3(1)
C(23)	17(1)	32(1)	21(1)	0(1)	-2(1)	-3(1)
C(24)	17(1)	28(1)	17(1)	2(1)	0(1)	4(1)
C(25)	18(1)	20(1)	16(1)	-2(1)	2(1)	2(1)
C(26)	18(1)	20(1)	16(1)	-2(1)	-2(1)	1(1)

C(27)	25(1)	18(1)	13(1)	-2(1)	-4(1)	4(1)

Table S4. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **M**.

	x	y	z	U(eq)
H(1)	1449	13049	5092	35
H(4)	3924	9022	5160	24
H(5)	5432	8041	5480	26
H(6)	5910	8006	6698	22
H(8)	3343	9908	7294	20
H(10)	6584	9770	8976	25
H(13)	3428	9950	9604	22
H(14)	1968	8809	9957	25
H(15)	1287	6390	9340	23
H(17)	3496	6344	7946	19
H(19)	1845	359	8218	26
H(22)	156	3462	6691	27
H(23)	-479	5196	5752	28
H(24)	141	8015	5486	25
H(26)	2122	7324	7071	22

Table S5. Torsion angles [°] for **M**.

C(27)-N(1)-C(1)-O(1)	-0.3(2)
C(2)-O(1)-C(1)-N(1)	0.1(2)
C(1)-O(1)-C(2)-C(27)	0.13(18)
C(1)-O(1)-C(2)-C(3)	175.03(15)
C(27)-C(2)-C(3)-C(8)	-75.4(3)
O(1)-C(2)-C(3)-C(8)	111.75(17)
C(27)-C(2)-C(3)-C(4)	108.7(2)
O(1)-C(2)-C(3)-C(4)	-64.2(2)
C(8)-C(3)-C(4)-C(5)	1.2(2)
C(2)-C(3)-C(4)-C(5)	177.23(15)
C(3)-C(4)-C(5)-C(6)	-0.8(2)
C(4)-C(5)-C(6)-C(7)	0.2(2)
C(5)-C(6)-C(7)-C(8)	0.0(2)
C(5)-C(6)-C(7)-C(9)	-176.38(15)
C(4)-C(3)-C(8)-C(7)	-1.0(2)
C(2)-C(3)-C(8)-C(7)	-176.88(15)
C(6)-C(7)-C(8)-C(3)	0.4(2)
C(9)-C(7)-C(8)-C(3)	176.72(15)
C(10)-N(2)-C(9)-C(11)	-1.09(18)
C(10)-N(2)-C(9)-C(7)	176.37(15)
C(8)-C(7)-C(9)-C(11)	32.5(3)
C(6)-C(7)-C(9)-C(11)	-151.20(18)
C(8)-C(7)-C(9)-N(2)	-144.26(15)
C(6)-C(7)-C(9)-N(2)	32.1(2)
C(9)-N(2)-C(10)-O(2)	0.76(19)
C(11)-O(2)-C(10)-N(2)	-0.14(19)
N(2)-C(9)-C(11)-O(2)	1.04(18)
C(7)-C(9)-C(11)-O(2)	-175.93(16)
N(2)-C(9)-C(11)-C(12)	-173.36(17)
C(7)-C(9)-C(11)-C(12)	9.7(3)
C(10)-O(2)-C(11)-C(9)	-0.57(17)
C(10)-O(2)-C(11)-C(12)	174.92(14)
C(9)-C(11)-C(12)-C(17)	38.7(3)
O(2)-C(11)-C(12)-C(17)	-135.32(15)
C(9)-C(11)-C(12)-C(13)	-145.62(19)

O(2)-C(11)-C(12)-C(13)	40.4(2)
C(17)-C(12)-C(13)-C(14)	0.3(2)
C(11)-C(12)-C(13)-C(14)	-175.36(15)
C(12)-C(13)-C(14)-C(15)	1.0(2)
C(13)-C(14)-C(15)-C(16)	-0.9(2)
C(14)-C(15)-C(16)-C(17)	-0.6(2)
C(14)-C(15)-C(16)-C(18)	172.37(14)
C(13)-C(12)-C(17)-C(16)	-1.8(2)
C(11)-C(12)-C(17)-C(16)	173.98(14)
C(15)-C(16)-C(17)-C(12)	2.0(2)
C(18)-C(16)-C(17)-C(12)	-171.26(14)
C(19)-N(3)-C(18)-C(20)	-0.30(17)
C(19)-N(3)-C(18)-C(16)	-177.14(14)
C(17)-C(16)-C(18)-C(20)	-78.8(2)
C(15)-C(16)-C(18)-C(20)	108.1(2)
C(17)-C(16)-C(18)-N(3)	97.09(17)
C(15)-C(16)-C(18)-N(3)	-76.0(2)
C(18)-N(3)-C(19)-O(3)	-0.11(18)
C(20)-O(3)-C(19)-N(3)	0.46(18)
N(3)-C(18)-C(20)-O(3)	0.58(17)
C(16)-C(18)-C(20)-O(3)	176.80(16)
N(3)-C(18)-C(20)-C(21)	178.11(17)
C(16)-C(18)-C(20)-C(21)	-5.7(3)
C(19)-O(3)-C(20)-C(18)	-0.62(17)
C(19)-O(3)-C(20)-C(21)	-178.70(13)
C(18)-C(20)-C(21)-C(22)	-148.82(19)
O(3)-C(20)-C(21)-C(22)	28.6(2)
C(18)-C(20)-C(21)-C(26)	31.5(3)
O(3)-C(20)-C(21)-C(26)	-151.16(15)
C(26)-C(21)-C(22)-C(23)	-1.5(2)
C(20)-C(21)-C(22)-C(23)	178.83(15)
C(21)-C(22)-C(23)-C(24)	-0.2(3)
C(22)-C(23)-C(24)-C(25)	2.1(2)
C(23)-C(24)-C(25)-C(26)	-2.2(2)
C(23)-C(24)-C(25)-C(27)	173.70(15)
C(24)-C(25)-C(26)-C(21)	0.5(2)
C(27)-C(25)-C(26)-C(21)	-175.12(15)

C(22)-C(21)-C(26)-C(25)	1.3(2)
C(20)-C(21)-C(26)-C(25)	-178.98(15)
O(1)-C(2)-C(27)-N(1)	-0.29(18)
C(3)-C(2)-C(27)-N(1)	-173.57(18)
O(1)-C(2)-C(27)-C(25)	170.06(16)
C(3)-C(2)-C(27)-C(25)	-3.2(3)
C(1)-N(1)-C(27)-C(2)	0.33(18)
C(1)-N(1)-C(27)-C(25)	-171.61(15)
C(26)-C(25)-C(27)-C(2)	30.8(3)
C(24)-C(25)-C(27)-C(2)	-144.92(18)
C(26)-C(25)-C(27)-N(1)	-159.64(15)
C(24)-C(25)-C(27)-N(1)	24.7(2)

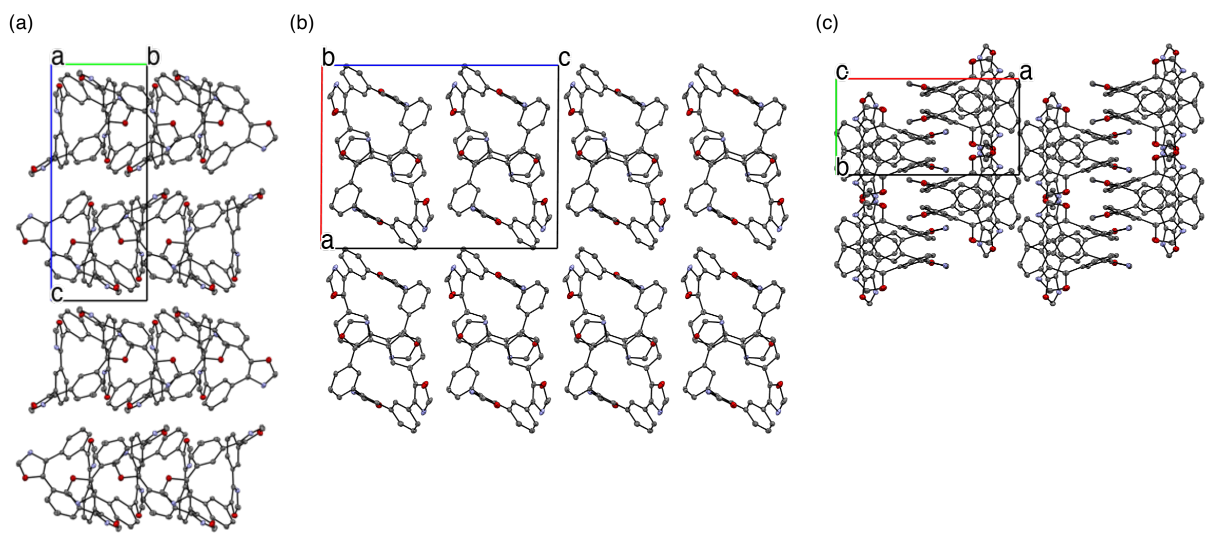

Figure S8. Molecular packing along a, b, and c axis, respectively.

4. DSC Curve

Differential scanning calorimetry (DSC) data were obtained using a DSC TA instrument Q20.

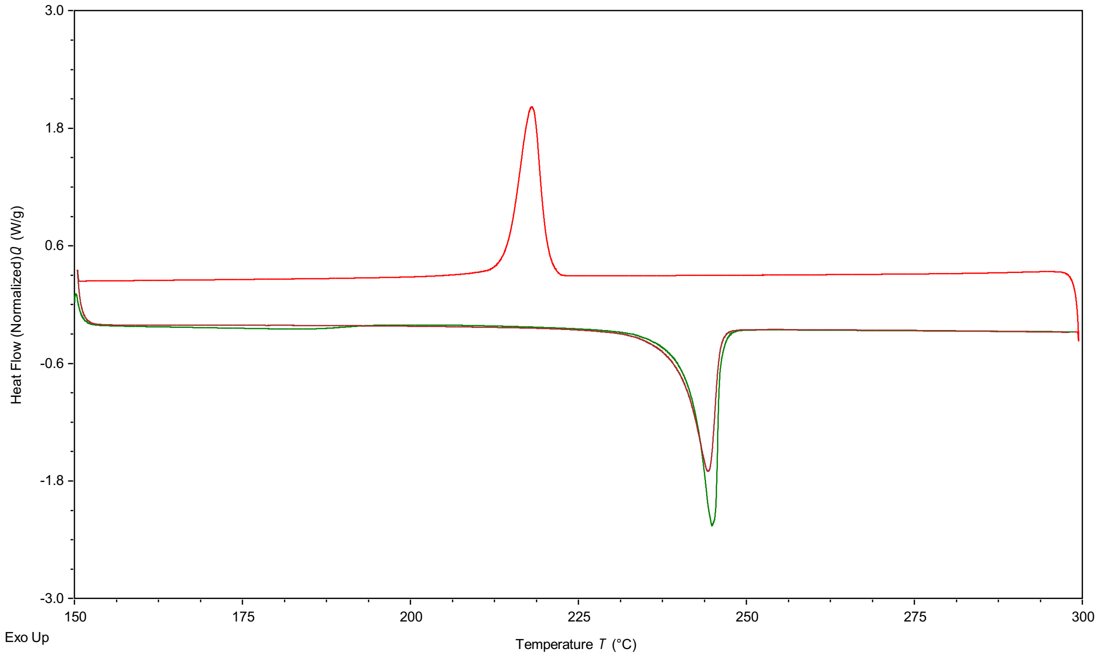

Figure S9. DSC heating-cooling-heating curves of **M** (heating rate: 10 °C/min).

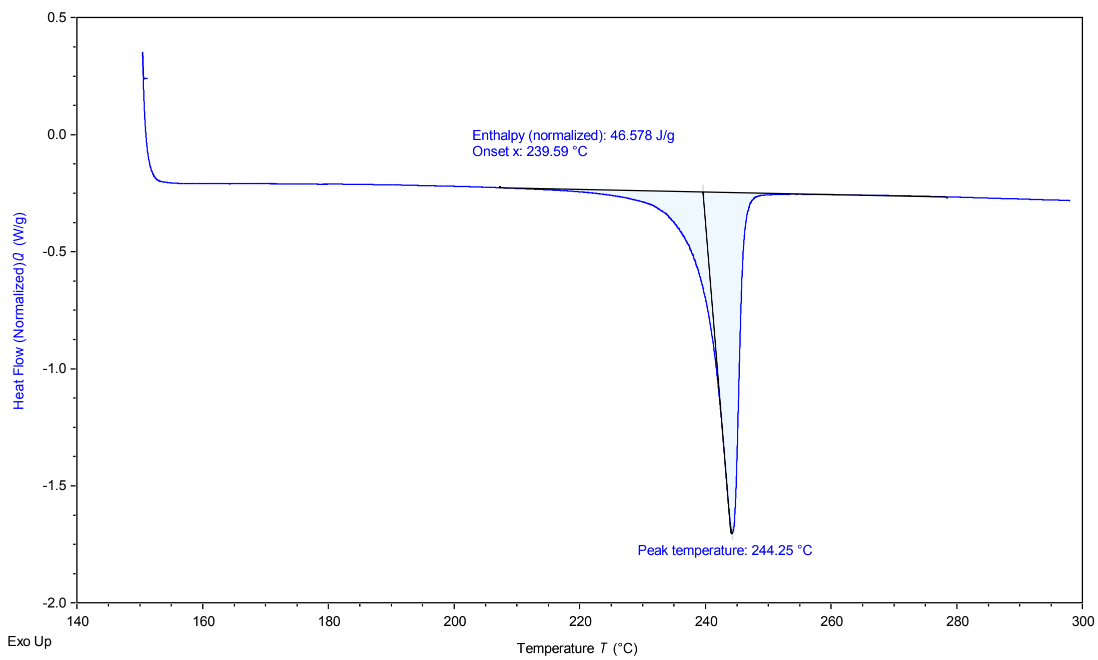

Figure S10. DSC heating curve of **M** (heating rate: 10 °C/min).

5. Hirshfeld Analysis

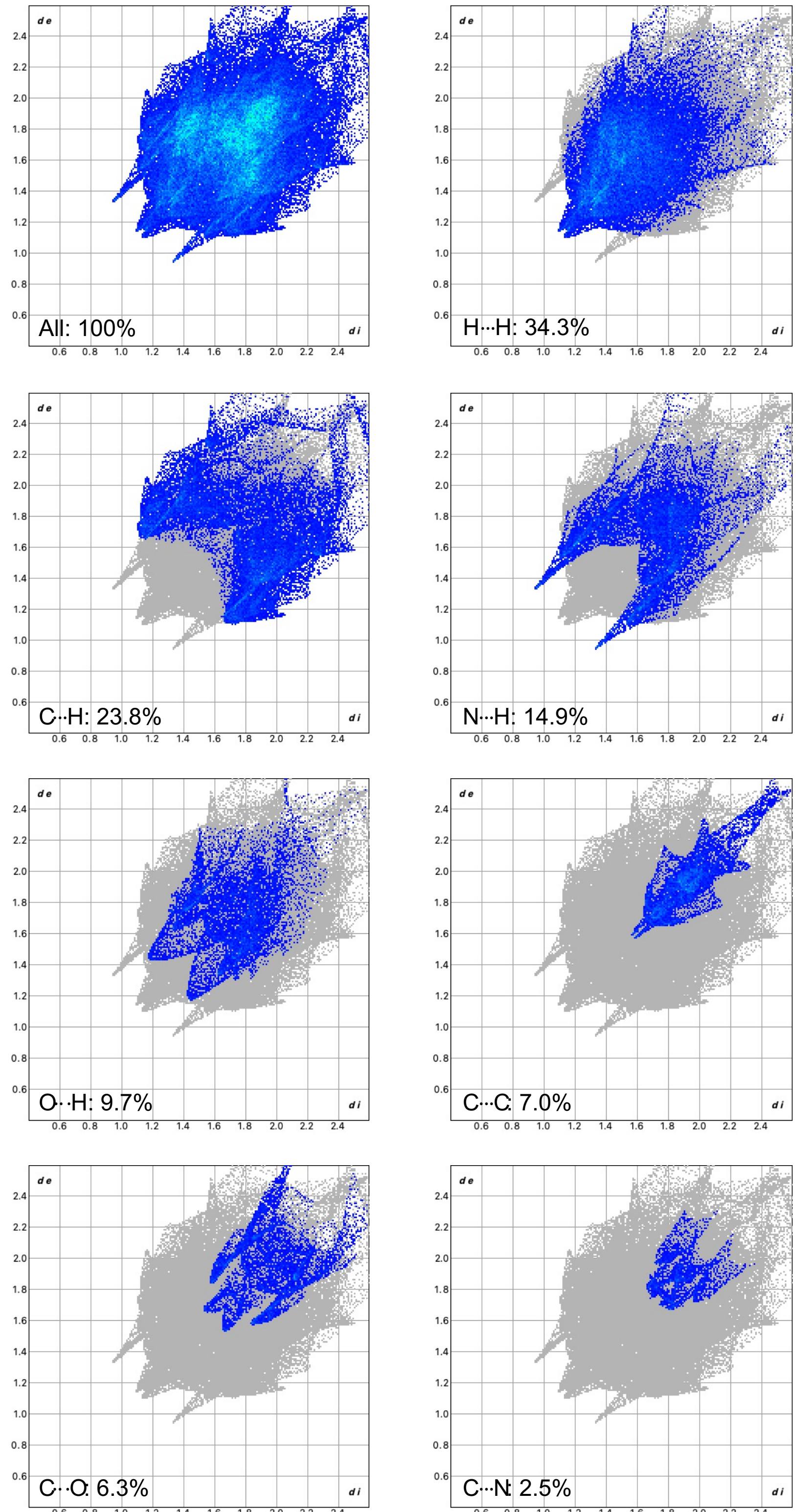

Figure S11. 2D fingerprint plots of **M** calculated from the Hirshfeld surface analysis using CrystalExplorer.