

Small Molecule X-ray Facility School Of Chemistry



Structure Report

Filename: TCD798

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Group: Meegan

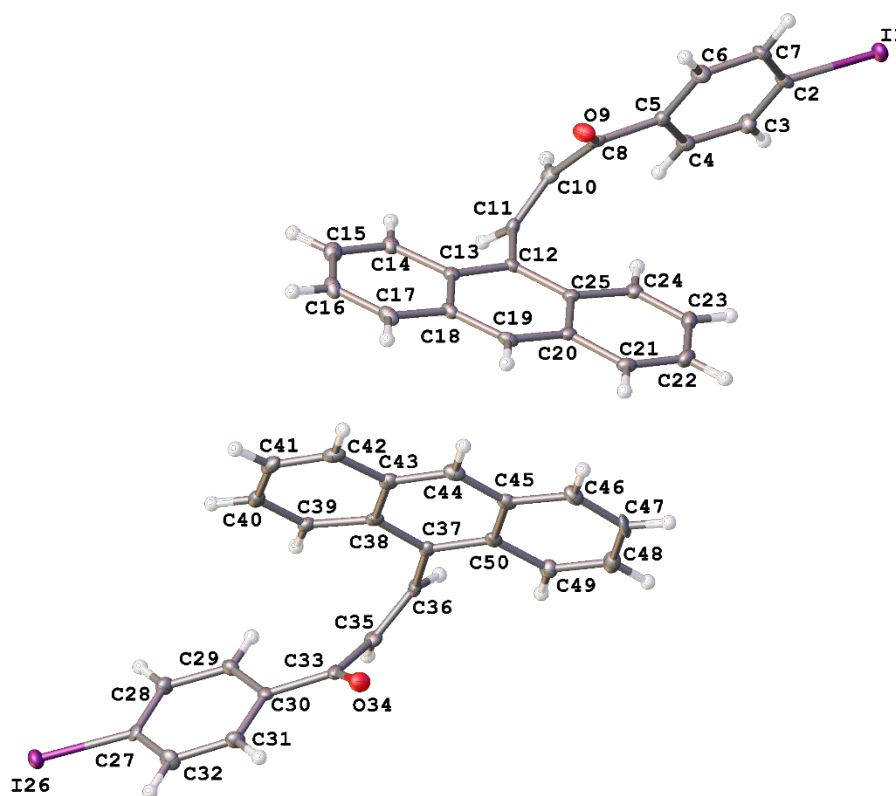


Fig. 1. Asymmetric unit of TCD798 with two independent molecules. Atomic displacement shown at 50% probability.

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Crystal Structure Report for TCD798

A specimen of $\text{C}_{23}\text{H}_{15}\text{IO}$, approximate dimensions 0.190 mm x 0.220 mm x 0.290 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K with an Oxford Cryosystems low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 2405 frames were collected. The total exposure time was 2.67 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using an orthorhombic unit cell yielded a total of 55052 reflections to a maximum θ angle of 27.18° (0.78 Å resolution), of which 7641 were independent (average redundancy 7.205, completeness = 99.6%, $R_{\text{int}} = 2.17\%$, $R_{\text{sig}} = 1.37\%$) and 7439 (97.36%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 10.8908(4)$ Å, $b = 10.4764(4)$ Å, $c = 30.3908(12)$ Å, volume = $3467.5(2)$ Å³, are based upon the refinement of the XYZ-centroids of 9798 reflections above $20\sigma(I)$ with $5.362^\circ < 2\theta < 54.31^\circ$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.919. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6848 and 0.7455.

Using Olex2, the structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimisation, using the space group $Pca2_1$, with $Z = 8$ for the formula unit, $\text{C}_{23}\text{H}_{15}\text{IO}$. The final anisotropic full-matrix least-squares refinement on F^2 with 446 variables converged at $R1 = 1.87\%$, for the observed data and $wR2 = 4.72\%$ for all data. The goodness-of-fit was 1.123. The largest peak in the final difference electron density synthesis was $0.846 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.579 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.071 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.664 g/cm^3 and $F(000)$, 1712 e^- .

Refinement Note: Flack test results are ambiguous.

References: see CIF.

Table 1: Data collection details for TCD798.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Phi	41.330	8.00	0.00	0.00	54.76	1.00	180	4.00	0.71073	50	20.0	100
Phi	41.330	27.72	209.72	0.00	54.76	0.40	900	4.00	0.71073	50	20.0	100
Omega	41.330	28.82	211.07	131.02	54.76	0.40	425	4.00	0.71073	50	20.0	100
Phi	41.330	27.72	209.72	0.00	54.76	0.40	900	4.00	0.71073	50	20.0	100

Crystal Data for C₂₃H₁₅IO (M = 434.25 g/mol): orthorhombic, space group Pca2₁ (no. 29), a = 10.8908(4) Å, b = 10.4764(4) Å, c = 30.3908(12) Å, V = 3467.5(2) Å³, Z = 8, T = 100(2) K, μ (MoK α) = 1.854 mm⁻¹, D_{calc} = 1.664 g/cm³, 55052 reflections measured ($5.396^\circ \leq 2\theta \leq 54.364^\circ$), 7641 unique (R_{int} = 0.0217, R_{sigma} = 0.0137) which were used in all calculations. The final R_1 was 0.0187 ($I > 2\sigma(I)$) and wR_2 was 0.0472 (all data).

Table 2. Crystal data and structure refinement for tcd798.

Identification code	tcd798	
Empirical formula	C ₂₃ H ₁₅ IO	
Formula weight	434.25	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pca2 ₁	
Unit cell dimensions	a = 10.8908(4) Å	α = 90°.
	b = 10.4764(4) Å	β = 90°.
	c = 30.3908(12) Å	γ = 90°.
Volume	3467.5(2) Å ³	
Z	8	
Density (calculated)	1.664 Mg/m ³	
Absorption coefficient	1.854 mm ⁻¹	
F(000)	1712	
Crystal size	0.29 x 0.22 x 0.19 mm ³	
Theta range for data collection	2.698 to 27.182°.	
Index ranges	-13 ≤ h ≤ 14, -13 ≤ k ≤ 13, -37 ≤ l ≤ 39	
Reflections collected	55052	
Independent reflections	7641 [R(int) = 0.0217]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7455 and 0.6848	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7641 / 1 / 446	
Goodness-of-fit on F ²	1.123	
Final R indices [I > 2σ(I)]	R1 = 0.0187, wR2 = 0.0467	
R indices (all data)	R1 = 0.0195, wR2 = 0.0472	
Absolute structure parameter	0.323(18)	
Largest diff. peak and hole	0.846 and -0.579 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd798. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	3633(1)	15864(1)	4711(1)	17(1)
O(9)	6194(3)	9807(3)	4293(1)	17(1)
C(2)	4104(4)	13947(4)	4558(1)	14(1)
C(3)	3360(4)	13273(4)	4281(1)	15(1)
C(4)	3676(3)	12028(4)	4177(2)	14(1)
C(5)	4746(3)	11483(3)	4350(1)	12(1)
C(6)	5467(3)	12194(3)	4637(1)	15(1)
C(7)	5147(3)	13430(3)	4754(1)	14(1)
C(8)	5147(4)	10162(4)	4224(2)	11(1)
C(10)	4212(3)	9324(4)	4010(1)	12(1)
C(11)	4454(3)	8529(3)	3679(1)	11(1)
C(12)	5624(3)	8363(3)	3434(1)	10(1)
C(13)	6123(3)	7127(3)	3400(1)	11(1)
C(14)	5580(3)	6038(3)	3606(1)	13(1)
C(15)	6109(5)	4857(4)	3584(2)	16(1)
C(16)	7242(4)	4696(4)	3354(2)	17(1)
C(17)	7786(4)	5689(4)	3152(1)	16(1)
C(18)	7256(3)	6934(3)	3163(1)	12(1)
C(19)	7811(3)	7972(4)	2957(1)	13(1)
C(20)	7311(3)	9192(3)	2972(1)	11(1)
C(21)	7874(4)	10229(4)	2748(2)	13(1)
C(22)	7386(4)	11437(4)	2770(1)	16(1)
C(23)	6284(4)	11646(4)	3014(1)	15(1)
C(24)	5710(4)	10671(4)	3224(1)	12(1)
C(25)	6189(3)	9402(4)	3218(1)	11(1)
I(26)	1177(1)	-816(1)	298(1)	17(1)
O(34)	3763(3)	5224(3)	716(1)	17(1)
C(27)	1643(4)	1066(3)	443(1)	12(1)
C(28)	923(4)	1765(4)	738(1)	15(1)
C(29)	1247(3)	3018(4)	840(2)	13(1)
C(30)	2302(3)	3557(3)	661(1)	11(1)
C(31)	3025(3)	2851(3)	371(1)	15(1)
C(32)	2695(3)	1611(3)	257(1)	17(1)

C(33)	2709(4)	4878(4)	783(2)	13(1)
C(35)	1786(4)	5741(3)	985(1)	13(1)
C(36)	2004(3)	6566(3)	1311(1)	12(1)
C(37)	3146(3)	6718(3)	1570(1)	11(1)
C(38)	3697(4)	5670(4)	1787(1)	12(1)
C(39)	3198(4)	4408(4)	1773(1)	13(1)
C(40)	3787(4)	3404(4)	1962(1)	16(1)
C(41)	4915(4)	3588(4)	2188(1)	17(1)
C(42)	5389(4)	4767(4)	2236(2)	16(1)
C(43)	4801(4)	5861(4)	2040(1)	14(1)
C(44)	5303(3)	7080(4)	2080(1)	15(1)
C(45)	4769(3)	8124(3)	1866(1)	13(1)
C(46)	5320(4)	9360(4)	1889(1)	16(1)
C(47)	4807(4)	10371(4)	1668(2)	18(1)
C(48)	3709(5)	10210(4)	1426(2)	18(1)
C(49)	3157(4)	9045(3)	1399(1)	14(1)
C(50)	3668(3)	7952(3)	1613(1)	12(1)

Table 4. Bond lengths [Å] and angles [°] for *tcd798*.

I(1)-C(2)	2.125(4)	C(21)-C(22)	1.374(6)
O(9)-C(8)	1.218(5)	C(22)-H(22)	0.9500
C(2)-C(3)	1.365(5)	C(22)-C(23)	1.427(6)
C(2)-C(7)	1.393(5)	C(23)-H(23)	0.9500
C(3)-H(3)	0.9500	C(23)-C(24)	1.357(5)
C(3)-C(4)	1.386(5)	C(24)-H(24)	0.9500
C(4)-H(4)	0.9500	C(24)-C(25)	1.428(5)
C(4)-C(5)	1.401(5)	I(26)-C(27)	2.083(4)
C(5)-C(6)	1.391(5)	O(34)-C(33)	1.220(6)
C(5)-C(8)	1.501(5)	C(27)-C(28)	1.398(5)
C(6)-H(6)	0.9500	C(27)-C(32)	1.399(5)
C(6)-C(7)	1.387(5)	C(28)-H(28)	0.9500
C(7)-H(7)	0.9500	C(28)-C(29)	1.395(5)
C(8)-C(10)	1.493(6)	C(29)-H(29)	0.9500
C(10)-H(10)	0.9500	C(29)-C(30)	1.392(5)
C(10)-C(11)	1.332(5)	C(30)-C(31)	1.394(5)
C(11)-H(11)	0.9500	C(30)-C(33)	1.500(5)
C(11)-C(12)	1.486(5)	C(31)-H(31)	0.9500
C(12)-C(13)	1.409(5)	C(31)-C(32)	1.391(5)
C(12)-C(25)	1.412(5)	C(32)-H(32)	0.9500
C(13)-C(14)	1.428(5)	C(33)-C(35)	1.485(6)
C(13)-C(18)	1.443(5)	C(35)-H(35)	0.9500
C(14)-H(14)	0.9500	C(35)-C(36)	1.337(5)
C(14)-C(15)	1.366(6)	C(36)-H(36)	0.9500
C(15)-H(15)	0.9500	C(36)-C(37)	1.480(5)
C(15)-C(16)	1.428(7)	C(37)-C(38)	1.415(5)
C(16)-H(16)	0.9500	C(37)-C(50)	1.418(5)
C(16)-C(17)	1.345(6)	C(38)-C(39)	1.430(5)
C(17)-H(17)	0.9500	C(38)-C(43)	1.441(6)
C(17)-C(18)	1.427(5)	C(39)-H(39)	0.9500
C(18)-C(19)	1.391(5)	C(39)-C(40)	1.359(5)
C(19)-H(19)	0.9500	C(40)-H(40)	0.9500
C(19)-C(20)	1.390(5)	C(40)-C(41)	1.420(6)
C(20)-C(21)	1.421(5)	C(41)-H(41)	0.9500
C(20)-C(25)	1.449(5)	C(41)-C(42)	1.346(6)
C(21)-H(21)	0.9500	C(42)-H(42)	0.9500

C(42)-C(43)	1.442(6)	C(11)-C(10)-H(10)	117.9
C(43)-C(44)	1.395(5)	C(10)-C(11)-H(11)	115.8
C(44)-H(44)	0.9500	C(10)-C(11)-C(12)	128.5(3)
C(44)-C(45)	1.398(5)	C(12)-C(11)-H(11)	115.8
C(45)-C(46)	1.429(5)	C(13)-C(12)-C(11)	118.3(3)
C(45)-C(50)	1.436(5)	C(13)-C(12)-C(25)	120.5(3)
C(46)-H(46)	0.9500	C(25)-C(12)-C(11)	121.1(3)
C(46)-C(47)	1.373(6)	C(12)-C(13)-C(14)	122.9(3)
C(47)-H(47)	0.9500	C(12)-C(13)-C(18)	119.6(3)
C(47)-C(48)	1.413(7)	C(14)-C(13)-C(18)	117.5(3)
C(48)-H(48)	0.9500	C(13)-C(14)-H(14)	119.1
C(48)-C(49)	1.363(6)	C(15)-C(14)-C(13)	121.8(4)
C(49)-H(49)	0.9500	C(15)-C(14)-H(14)	119.1
C(49)-C(50)	1.430(5)	C(14)-C(15)-H(15)	120.2
		C(14)-C(15)-C(16)	119.7(4)
C(3)-C(2)-I(1)	118.7(3)	C(16)-C(15)-H(15)	120.2
C(3)-C(2)-C(7)	123.2(4)	C(15)-C(16)-H(16)	119.6
C(7)-C(2)-I(1)	118.1(3)	C(17)-C(16)-C(15)	120.8(4)
C(2)-C(3)-H(3)	120.7	C(17)-C(16)-H(16)	119.6
C(2)-C(3)-C(4)	118.6(4)	C(16)-C(17)-H(17)	119.4
C(4)-C(3)-H(3)	120.7	C(16)-C(17)-C(18)	121.2(4)
C(3)-C(4)-H(4)	119.8	C(18)-C(17)-H(17)	119.4
C(3)-C(4)-C(5)	120.3(4)	C(17)-C(18)-C(13)	119.0(3)
C(5)-C(4)-H(4)	119.8	C(19)-C(18)-C(13)	119.1(3)
C(4)-C(5)-C(8)	121.5(3)	C(19)-C(18)-C(17)	121.9(3)
C(6)-C(5)-C(4)	119.2(3)	C(18)-C(19)-H(19)	118.9
C(6)-C(5)-C(8)	119.4(3)	C(20)-C(19)-C(18)	122.3(3)
C(5)-C(6)-H(6)	119.4	C(20)-C(19)-H(19)	118.9
C(7)-C(6)-C(5)	121.3(3)	C(19)-C(20)-C(21)	121.3(4)
C(7)-C(6)-H(6)	119.4	C(19)-C(20)-C(25)	119.1(3)
C(2)-C(7)-H(7)	121.4	C(21)-C(20)-C(25)	119.7(3)
C(6)-C(7)-C(2)	117.3(3)	C(20)-C(21)-H(21)	119.5
C(6)-C(7)-H(7)	121.4	C(22)-C(21)-C(20)	120.9(4)
O(9)-C(8)-C(5)	120.6(4)	C(22)-C(21)-H(21)	119.5
O(9)-C(8)-C(10)	122.3(4)	C(21)-C(22)-H(22)	120.3
C(10)-C(8)-C(5)	117.1(3)	C(21)-C(22)-C(23)	119.5(4)
C(8)-C(10)-H(10)	117.9	C(23)-C(22)-H(22)	120.3
C(11)-C(10)-C(8)	124.1(4)	C(22)-C(23)-H(23)	119.5

C(24)-C(23)-C(22)	121.0(4)	C(37)-C(38)-C(39)	122.8(4)
C(24)-C(23)-H(23)	119.5	C(37)-C(38)-C(43)	119.7(3)
C(23)-C(24)-H(24)	119.1	C(39)-C(38)-C(43)	117.5(3)
C(23)-C(24)-C(25)	121.8(4)	C(38)-C(39)-H(39)	119.2
C(25)-C(24)-H(24)	119.1	C(40)-C(39)-C(38)	121.5(4)
C(12)-C(25)-C(20)	119.4(3)	C(40)-C(39)-H(39)	119.2
C(12)-C(25)-C(24)	123.5(3)	C(39)-C(40)-H(40)	119.7
C(24)-C(25)-C(20)	117.1(3)	C(39)-C(40)-C(41)	120.5(4)
C(28)-C(27)-I(26)	119.6(3)	C(41)-C(40)-H(40)	119.7
C(28)-C(27)-C(32)	120.3(3)	C(40)-C(41)-H(41)	119.7
C(32)-C(27)-I(26)	120.0(3)	C(42)-C(41)-C(40)	120.6(4)
C(27)-C(28)-H(28)	120.2	C(42)-C(41)-H(41)	119.7
C(29)-C(28)-C(27)	119.6(4)	C(41)-C(42)-H(42)	119.5
C(29)-C(28)-H(28)	120.2	C(41)-C(42)-C(43)	120.9(4)
C(28)-C(29)-H(29)	119.9	C(43)-C(42)-H(42)	119.5
C(30)-C(29)-C(28)	120.3(4)	C(42)-C(43)-C(38)	118.7(4)
C(30)-C(29)-H(29)	119.9	C(44)-C(43)-C(38)	120.0(3)
C(29)-C(30)-C(31)	119.9(3)	C(44)-C(43)-C(42)	121.2(4)
C(29)-C(30)-C(33)	121.5(3)	C(43)-C(44)-H(44)	119.6
C(31)-C(30)-C(33)	118.6(3)	C(43)-C(44)-C(45)	120.8(3)
C(30)-C(31)-H(31)	119.8	C(45)-C(44)-H(44)	119.6
C(32)-C(31)-C(30)	120.5(3)	C(44)-C(45)-C(46)	120.8(3)
C(32)-C(31)-H(31)	119.8	C(44)-C(45)-C(50)	119.9(3)
C(27)-C(32)-H(32)	120.3	C(46)-C(45)-C(50)	119.3(3)
C(31)-C(32)-C(27)	119.5(3)	C(45)-C(46)-H(46)	119.9
C(31)-C(32)-H(32)	120.3	C(47)-C(46)-C(45)	120.3(4)
O(34)-C(33)-C(30)	120.7(4)	C(47)-C(46)-H(46)	119.9
O(34)-C(33)-C(35)	121.6(4)	C(46)-C(47)-H(47)	119.8
C(35)-C(33)-C(30)	117.6(4)	C(46)-C(47)-C(48)	120.5(4)
C(33)-C(35)-H(35)	117.3	C(48)-C(47)-H(47)	119.8
C(36)-C(35)-C(33)	125.5(4)	C(47)-C(48)-H(48)	119.6
C(36)-C(35)-H(35)	117.3	C(49)-C(48)-C(47)	120.7(4)
C(35)-C(36)-H(36)	116.1	C(49)-C(48)-H(48)	119.6
C(35)-C(36)-C(37)	127.8(3)	C(48)-C(49)-H(49)	119.4
C(37)-C(36)-H(36)	116.1	C(48)-C(49)-C(50)	121.2(4)
C(38)-C(37)-C(36)	121.4(3)	C(50)-C(49)-H(49)	119.4
C(38)-C(37)-C(50)	119.6(3)	C(37)-C(50)-C(45)	119.9(3)
C(50)-C(37)-C(36)	119.0(3)	C(37)-C(50)-C(49)	122.1(3)

C(49)-C(50)-C(45)

117.9(3)

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd798. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	22(1)	12(1)	17(1)	-3(1)	2(1)	2(1)
O(9)	12(2)	21(2)	16(2)	-3(1)	-1(1)	5(1)
C(2)	17(1)	16(1)	11(1)	-6(1)	0(1)	-1(1)
C(3)	14(2)	14(2)	17(2)	1(2)	-2(2)	2(1)
C(4)	12(2)	16(2)	12(2)	-1(2)	-2(1)	-1(1)
C(5)	13(2)	11(2)	11(2)	0(1)	2(1)	1(1)
C(6)	12(2)	18(2)	15(2)	-1(1)	-3(1)	2(1)
C(7)	17(1)	16(1)	11(1)	-6(1)	0(1)	-1(1)
C(8)	11(2)	16(2)	6(2)	3(1)	1(2)	0(1)
C(10)	9(2)	15(2)	14(2)	3(1)	-1(1)	3(1)
C(11)	11(2)	9(2)	14(2)	2(1)	1(1)	0(1)
C(12)	11(2)	12(2)	9(2)	-3(1)	-1(1)	0(1)
C(13)	11(2)	13(2)	9(2)	-1(1)	-2(1)	-1(1)
C(14)	12(2)	13(2)	14(2)	-1(1)	-3(1)	-1(1)
C(15)	15(2)	16(2)	19(2)	0(2)	-3(2)	-2(2)
C(16)	16(2)	12(2)	22(2)	-3(2)	-8(2)	2(2)
C(17)	13(2)	18(2)	17(2)	-6(2)	-3(2)	4(1)
C(18)	12(2)	12(2)	12(2)	-4(1)	-3(1)	3(1)
C(19)	11(2)	18(2)	11(2)	-4(1)	1(1)	0(1)
C(20)	13(2)	14(2)	8(2)	0(1)	-1(1)	-1(1)
C(21)	12(2)	18(2)	10(2)	0(2)	0(2)	-2(1)
C(22)	19(2)	16(2)	14(2)	4(2)	-1(2)	-4(2)
C(23)	19(2)	12(2)	15(2)	1(1)	-4(2)	-1(2)
C(24)	13(2)	15(2)	10(2)	0(1)	-2(2)	1(1)
C(25)	11(2)	13(2)	8(2)	0(1)	-2(2)	1(1)
I(26)	22(1)	12(1)	17(1)	-2(1)	-2(1)	-2(1)
O(34)	13(2)	21(1)	18(2)	-1(1)	0(1)	-3(1)
C(27)	15(2)	9(1)	12(2)	4(1)	-1(1)	3(1)
C(28)	14(2)	15(2)	14(2)	1(1)	2(2)	-3(1)
C(29)	15(2)	13(2)	12(2)	-2(2)	1(1)	1(1)
C(30)	14(2)	12(2)	8(2)	0(1)	-2(1)	0(1)
C(31)	14(2)	18(2)	13(2)	-1(1)	2(1)	-2(1)
C(32)	13(2)	18(2)	20(2)	-4(2)	3(2)	2(1)

C(33)	18(2)	12(2)	9(2)	1(1)	-3(2)	-3(1)
C(35)	13(2)	15(2)	12(2)	2(1)	-4(1)	0(1)
C(36)	14(2)	8(2)	15(2)	2(1)	1(1)	0(1)
C(37)	11(2)	11(2)	10(2)	0(1)	4(1)	1(1)
C(38)	12(2)	16(2)	8(2)	0(1)	3(2)	2(1)
C(39)	14(2)	15(2)	9(2)	1(1)	0(2)	0(1)
C(40)	20(2)	13(2)	13(2)	1(1)	4(2)	0(2)
C(41)	20(2)	17(2)	14(2)	5(2)	3(2)	6(2)
C(42)	15(2)	24(2)	10(2)	1(2)	1(2)	4(2)
C(43)	12(2)	17(2)	12(2)	0(1)	3(1)	1(1)
C(44)	12(2)	22(2)	13(2)	-4(1)	-1(1)	0(1)
C(45)	10(2)	17(2)	12(2)	-3(1)	2(1)	-1(1)
C(46)	14(2)	18(2)	17(2)	-6(2)	0(2)	-3(1)
C(47)	22(2)	8(2)	25(2)	-5(2)	5(2)	-6(2)
C(48)	20(2)	14(2)	21(2)	1(2)	4(2)	2(2)
C(49)	13(2)	14(2)	16(2)	0(1)	0(2)	2(1)
C(50)	11(2)	14(2)	10(2)	-2(1)	4(1)	0(1)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd798.

	x	y	z	U(eq)
H(3)	2640	13651	4161	18
H(4)	3163	11541	3988	16
H(6)	6192	11826	4756	18
H(7)	5619	13904	4960	17
H(10)	3393	9359	4117	15
H(11)	3793	7999	3588	14
H(14)	4831	6138	3762	16
H(15)	5725	4147	3721	20
H(16)	7616	3878	3343	20
H(17)	8537	5559	3000	19
H(19)	8557	7842	2802	16
H(21)	8598	10086	2581	16
H(22)	7778	12127	2624	20
H(23)	5947	12482	3030	18
H(24)	4969	10837	3378	15
H(28)	217	1388	868	18
H(29)	745	3506	1032	16
H(31)	3748	3219	250	18
H(32)	3181	1138	55	20
H(35)	972	5704	873	16
H(36)	1347	7122	1386	15
H(39)	2438	4267	1627	15
H(40)	3441	2574	1943	19
H(41)	5338	2874	2306	20
H(42)	6122	4879	2401	20
H(44)	6016	7204	2255	18
H(46)	6045	9484	2057	20
H(47)	5192	11184	1677	22
H(48)	3352	10922	1281	22
H(49)	2419	8958	1235	17

Table 7. Torsion angles [°] for tcd798.

I(1)-C(2)-C(3)-C(4)	179.3(3)	C(17)-C(18)-C(19)-C(20)	-178.9(3)
I(1)-C(2)-C(7)-C(6)	-177.6(3)	C(18)-C(13)-C(14)-C(15)	0.5(6)
O(9)-C(8)-C(10)-C(11)	-38.7(7)	C(18)-C(19)-C(20)-C(21)	-177.9(4)
C(2)-C(3)-C(4)-C(5)	-0.8(6)	C(18)-C(19)-C(20)-C(25)	1.4(6)
C(3)-C(2)-C(7)-C(6)	3.9(6)	C(19)-C(20)-C(21)-C(22)	-179.0(4)
C(3)-C(4)-C(5)-C(6)	2.0(6)	C(19)-C(20)-C(25)-C(12)	-0.5(5)
C(3)-C(4)-C(5)-C(8)	-176.6(4)	C(19)-C(20)-C(25)-C(24)	-180.0(3)
C(4)-C(5)-C(6)-C(7)	-0.2(6)	C(20)-C(21)-C(22)-C(23)	-1.3(6)
C(4)-C(5)-C(8)-O(9)	163.2(4)	C(21)-C(20)-C(25)-C(12)	178.9(4)
C(4)-C(5)-C(8)-C(10)	-16.3(6)	C(21)-C(20)-C(25)-C(24)	-0.6(5)
C(5)-C(6)-C(7)-C(2)	-2.6(6)	C(21)-C(22)-C(23)-C(24)	-0.1(6)
C(5)-C(8)-C(10)-C(11)	140.8(4)	C(22)-C(23)-C(24)-C(25)	1.3(6)
C(6)-C(5)-C(8)-O(9)	-15.5(6)	C(23)-C(24)-C(25)-C(12)	179.7(4)
C(6)-C(5)-C(8)-C(10)	165.1(4)	C(23)-C(24)-C(25)-C(20)	-0.9(5)
C(7)-C(2)-C(3)-C(4)	-2.2(6)	C(25)-C(12)-C(13)-C(14)	-179.1(3)
C(8)-C(5)-C(6)-C(7)	178.5(4)	C(25)-C(12)-C(13)-C(18)	3.3(5)
C(8)-C(10)-C(11)-C(12)	-4.8(6)	C(25)-C(20)-C(21)-C(22)	1.7(6)
C(10)-C(11)-C(12)-C(13)	126.6(4)	I(26)-C(27)-C(28)-C(29)	179.0(3)
C(10)-C(11)-C(12)-C(25)	-56.9(5)	I(26)-C(27)-C(32)-C(31)	-177.3(3)
C(11)-C(12)-C(13)-C(14)	-2.5(5)	O(34)-C(33)-C(35)-C(36)	-38.7(7)
C(11)-C(12)-C(13)-C(18)	179.8(3)	C(27)-C(28)-C(29)-C(30)	-2.1(6)
C(11)-C(12)-C(25)-C(20)	-178.4(3)	C(28)-C(27)-C(32)-C(31)	0.5(6)
C(11)-C(12)-C(25)-C(24)	1.1(5)	C(28)-C(29)-C(30)-C(31)	1.4(6)
C(12)-C(13)-C(14)-C(15)	-177.2(4)	C(28)-C(29)-C(30)-C(33)	-176.8(4)
C(12)-C(13)-C(18)-C(17)	176.5(3)	C(29)-C(30)-C(31)-C(32)	0.3(6)
C(12)-C(13)-C(18)-C(19)	-2.3(5)	C(29)-C(30)-C(33)-O(34)	161.5(5)
C(13)-C(12)-C(25)-C(20)	-1.9(5)	C(29)-C(30)-C(33)-C(35)	-18.6(6)
C(13)-C(12)-C(25)-C(24)	177.6(3)	C(30)-C(31)-C(32)-C(27)	-1.2(6)
C(13)-C(14)-C(15)-C(16)	0.7(7)	C(30)-C(33)-C(35)-C(36)	141.3(4)
C(13)-C(18)-C(19)-C(20)	0.0(6)	C(31)-C(30)-C(33)-O(34)	-16.7(6)
C(14)-C(13)-C(18)-C(17)	-1.2(5)	C(31)-C(30)-C(33)-C(35)	163.2(4)
C(14)-C(13)-C(18)-C(19)	179.9(3)	C(32)-C(27)-C(28)-C(29)	1.2(6)
C(14)-C(15)-C(16)-C(17)	-1.1(7)	C(33)-C(30)-C(31)-C(32)	178.5(4)
C(15)-C(16)-C(17)-C(18)	0.3(6)	C(33)-C(35)-C(36)-C(37)	-6.6(6)
C(16)-C(17)-C(18)-C(13)	0.8(6)	C(35)-C(36)-C(37)-C(38)	-53.7(5)
C(16)-C(17)-C(18)-C(19)	179.7(4)	C(35)-C(36)-C(37)-C(50)	128.8(4)

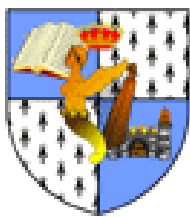
C(36)-C(37)-C(38)-C(39)	0.1(5)	C(42)-C(43)-C(44)-C(45)	176.0(4)
C(36)-C(37)-C(38)-C(43)	-179.3(3)	C(43)-C(38)-C(39)-C(40)	-4.7(5)
C(36)-C(37)-C(50)-C(45)	179.4(3)	C(43)-C(44)-C(45)-C(46)	-176.7(4)
C(36)-C(37)-C(50)-C(49)	-2.8(5)	C(43)-C(44)-C(45)-C(50)	2.0(5)
C(37)-C(38)-C(39)-C(40)	175.9(4)	C(44)-C(45)-C(46)-C(47)	178.2(4)
C(37)-C(38)-C(43)-C(42)	-176.1(4)	C(44)-C(45)-C(50)-C(37)	-1.9(5)
C(37)-C(38)-C(43)-C(44)	1.9(6)	C(44)-C(45)-C(50)-C(49)	-179.8(3)
C(38)-C(37)-C(50)-C(45)	1.8(5)	C(45)-C(46)-C(47)-C(48)	1.8(6)
C(38)-C(37)-C(50)-C(49)	179.6(3)	C(46)-C(45)-C(50)-C(37)	176.8(3)
C(38)-C(39)-C(40)-C(41)	1.0(6)	C(46)-C(45)-C(50)-C(49)	-1.1(5)
C(38)-C(43)-C(44)-C(45)	-2.0(6)	C(46)-C(47)-C(48)-C(49)	-1.6(7)
C(39)-C(38)-C(43)-C(42)	4.5(5)	C(47)-C(48)-C(49)-C(50)	0.0(7)
C(39)-C(38)-C(43)-C(44)	-177.5(4)	C(48)-C(49)-C(50)-C(37)	-176.5(4)
C(39)-C(40)-C(41)-C(42)	3.1(6)	C(48)-C(49)-C(50)-C(45)	1.4(6)
C(40)-C(41)-C(42)-C(43)	-3.3(6)	C(50)-C(37)-C(38)-C(39)	177.6(3)
C(41)-C(42)-C(43)-C(38)	-0.6(6)	C(50)-C(37)-C(38)-C(43)	-1.8(5)
C(41)-C(42)-C(43)-C(44)	-178.6(4)	C(50)-C(45)-C(46)-C(47)	-0.5(6)

Table 8. Hydrogen bonds for tcd798 [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(7)-H(7)...O(34)#1	0.95	2.56	3.457(5)	157
C(10)-H(10)...O(9)#2	0.95	2.60	3.516(5)	161

Symmetry transformations used to generate equivalent atoms:

#1 $-x+1, -y+2, z+1/2$ #2 $x-1/2, -y+2, z$



Small Molecule X-ray Facility School Of Chemistry



Structure Report

Filename: TCD654b

Submitted by: James McKeown

Reference: JB76 (yellow)

Group: Meegan

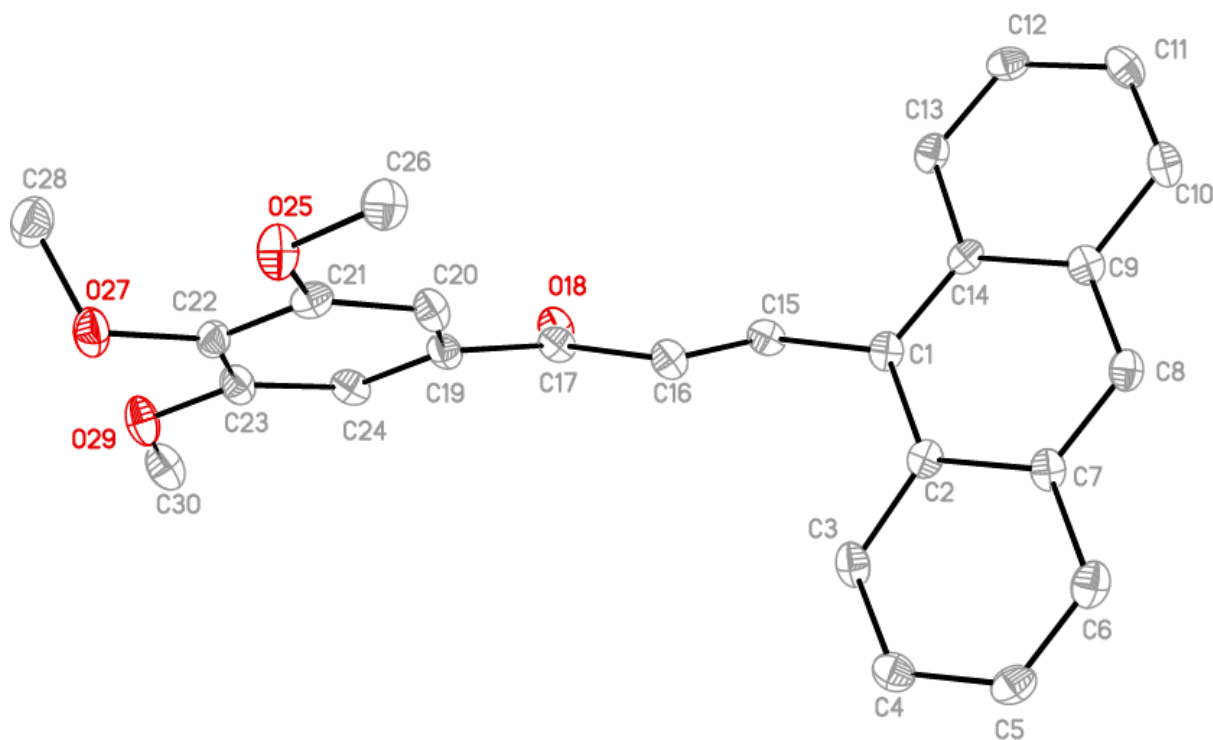


Fig. 1. Asymmetric unit of TCD654b with atomic displacement shown at 50% probability.
Hydrogen atoms omitted for clarity.

24/11/16

Author: Brendan Twamley

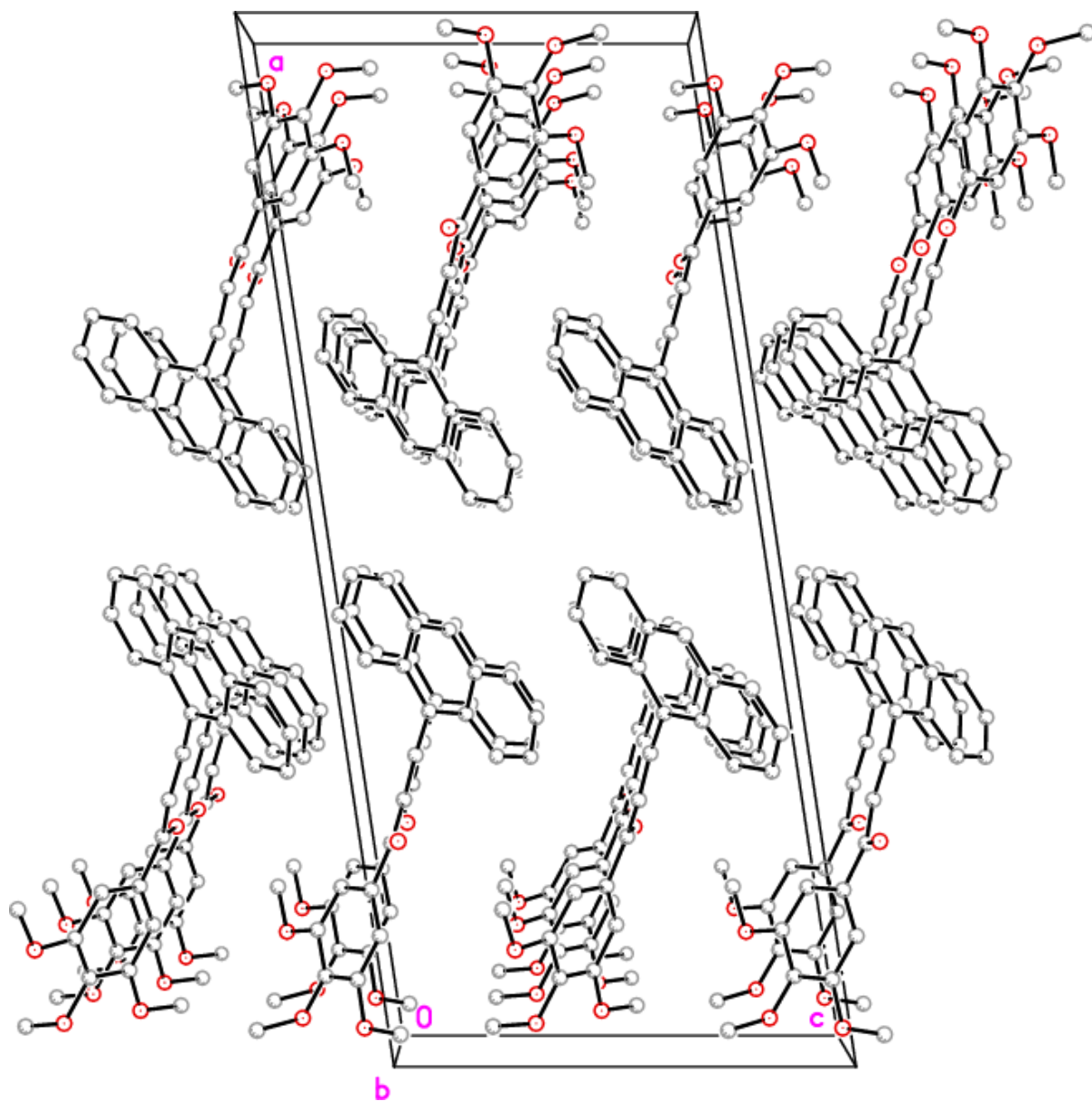


Fig. 2. Packing diagram of TCD654b viewed normal to the b-axis. Hydrogen atoms omitted for clarity.

Crystal Structure Report for TCD654b

A clear yellow block-like specimen of $\text{C}_{26}\text{H}_{22}\text{O}_4$, approximate dimensions 0.040 mm x 0.130 mm x 0.260 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K using an Oxford Cryosystems low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 874 frames were collected. The total exposure time was 10.14 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 18897 reflections to a maximum θ angle of 25.60° ($0.82 \text{ \AA}$ resolution), of which 3698 were independent (average redundancy 5.110, completeness = 99.4%, $R_{\text{int}} = 7.72\%$, $R_{\text{sig}} = 5.83\%$) and 2712 (73.34%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 29.099(3) \text{ \AA}$, $b = 5.4348(5) \text{ \AA}$, $c = 12.6086(11) \text{ \AA}$, $\beta = 98.582(3)^\circ$, volume = $1971.7(3) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 5325 reflections above $20 \sigma(I)$ with $5.663^\circ < 2\theta < 50.56^\circ$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.802. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.5974 and 0.7452.

The structure was solved using the Bruker APEX Software Package and refined with XL in Olex2, using the space group $P2_1/c$, with $Z = 4$ for the formula unit, $\text{C}_{26}\text{H}_{22}\text{O}_4$. The final anisotropic full-matrix least-squares refinement on F^2 with 274 variables converged at $R1 = 7.54\%$, for the observed data and $wR2 = 18.03\%$ for all data. The goodness-of-fit was 1.122. The largest peak in the final difference electron density synthesis was $0.282 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.334 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.076 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.342 g/cm^3 and $F(000)$, 840 e^- .

References: see CIF.

Table 1: Data collection details for TCD654b.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Phi	41.245	8.00	0.00	0.00	54.76	1.00	180	10.00	0.71073	50	20.0	100
Omega	41.245	21.70	205.57	153.00	54.76	0.80	209	50.00	0.71073	50	20.0	100
Omega	41.245	21.70	205.57	255.00	54.76	0.80	209	50.00	0.71073	50	20.0	100
Phi	41.245	21.70	14.64	50.80	54.76	0.80	123	50.00	0.71073	50	20.0	100
Phi	41.245	21.70	203.70	226.80	54.76	0.80	153	50.00	0.71073	50	20.0	100

Crystal Data for C₂₆H₂₂O₄ (M = 398.43 g/mol): monoclinic, space group P2₁/c (no. 14), a = 29.099(3) Å, b = 5.4348(5) Å, c = 12.6086(11) Å, β = 98.582(3)°, V = 1971.7(3) Å³, Z = 4, T = 100(2) K, μ (MoK α) = 0.090 mm⁻¹, D_{calc} = 1.342 g/cm³, 18897 reflections measured ($5.664^\circ \leq 2\theta \leq 51.192^\circ$), 3698 unique (R_{int} = 0.0772, R_{sigma} = 0.0583) which were used in all calculations. The final R_1 was 0.0754 ($I > 2\sigma(I)$) and wR_2 was 0.1803 (all data).

Table 2. Crystal data and structure refinement for tcd654b.

Identification code	tcd654b	
Empirical formula	C ₂₆ H ₂₂ O ₄	
Formula weight	398.43	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 29.099(3) Å	α = 90°.
	b = 5.4348(5) Å	β = 98.582(3)°.
	c = 12.6086(11) Å	γ = 90°.
Volume	1971.7(3) Å ³	
Z	4	
Density (calculated)	1.342 Mg/m ³	
Absorption coefficient	0.090 mm ⁻¹	
F(000)	840	
Crystal size	0.26 x 0.13 x 0.04 mm ³	
Theta range for data collection	2.832 to 25.596°.	
Index ranges	-35 ≤ h ≤ 35, -6 ≤ k ≤ 6, -15 ≤ l ≤ 15	
Reflections collected	18897	
Independent reflections	3698 [R(int) = 0.0772]	
Completeness to theta = 25.596°	99.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7452 and 0.5974	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3698 / 0 / 274	
Goodness-of-fit on F ²	1.122	
Final R indices [I > 2σ(I)]	R1 = 0.0754, wR2 = 0.1683	
R indices (all data)	R1 = 0.1046, wR2 = 0.1803	
Largest diff. peak and hole	0.282 and -0.334 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd654b. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
C(1)	3390(1)	3443(6)	6952(3)	14(1)
C(2)	3343(1)	5314(6)	7704(3)	14(1)
C(3)	2940(1)	5571(7)	8232(3)	19(1)
C(4)	2909(1)	7401(7)	8947(3)	20(1)
C(5)	3270(1)	9151(7)	9194(3)	20(1)
C(6)	3666(1)	8928(7)	8754(3)	20(1)
C(7)	3722(1)	6997(6)	8020(3)	16(1)
C(8)	4134(1)	6717(7)	7593(3)	17(1)
C(9)	4193(1)	4813(6)	6886(3)	16(1)
C(10)	4618(1)	4525(7)	6442(3)	21(1)
C(11)	4671(1)	2689(7)	5737(3)	22(1)
C(12)	4303(1)	1043(7)	5417(3)	19(1)
C(13)	3890(1)	1242(7)	5812(3)	18(1)
C(14)	3820(1)	3127(6)	6560(3)	14(1)
C(15)	2998(1)	1817(7)	6552(3)	16(1)
C(16)	2572(1)	2595(7)	6148(3)	17(1)
C(17)	2198(1)	782(6)	5789(3)	16(1)
O(18)	2255(1)	-1415(5)	5994(2)	20(1)
C(19)	1749(1)	1659(7)	5187(3)	17(1)
C(20)	1733(1)	3663(6)	4493(3)	18(1)
C(21)	1318(1)	4197(6)	3822(3)	17(1)
C(22)	920(1)	2785(7)	3899(3)	17(1)
C(23)	938(1)	877(7)	4639(3)	19(1)
C(24)	1356(1)	267(7)	5271(3)	17(1)
O(25)	1258(1)	6031(5)	3078(2)	21(1)
C(26)	1657(1)	7505(7)	2966(3)	22(1)
O(27)	510(1)	3363(5)	3253(2)	23(1)
C(28)	438(1)	1868(8)	2308(3)	28(1)
O(29)	525(1)	-302(5)	4676(2)	24(1)
C(30)	521(1)	-2211(7)	5457(3)	24(1)

Table 4. Bond lengths [Å] and angles [°] for tcd654b.

C(1)-C(2)	1.411(5)	C(21)-C(22)	1.405(5)
C(1)-C(14)	1.421(5)	C(21)-O(25)	1.362(4)
C(1)-C(15)	1.472(5)	C(22)-C(23)	1.391(5)
C(2)-C(3)	1.438(5)	C(22)-O(27)	1.377(4)
C(2)-C(7)	1.442(5)	C(23)-C(24)	1.390(5)
C(3)-H(3)	0.9500	C(23)-O(29)	1.368(4)
C(3)-C(4)	1.354(5)	C(24)-H(24)	0.9500
C(4)-H(4)	0.9500	O(25)-C(26)	1.434(4)
C(4)-C(5)	1.417(5)	C(26)-H(26A)	0.9800
C(5)-H(5)	0.9500	C(26)-H(26B)	0.9800
C(5)-C(6)	1.358(5)	C(26)-H(26C)	0.9800
C(6)-H(6)	0.9500	O(27)-C(28)	1.431(5)
C(6)-C(7)	1.424(5)	C(28)-H(28A)	0.9800
C(7)-C(8)	1.395(5)	C(28)-H(28B)	0.9800
C(8)-H(8)	0.9500	C(28)-H(28C)	0.9800
C(8)-C(9)	1.393(5)	O(29)-C(30)	1.431(5)
C(9)-C(10)	1.438(5)	C(30)-H(30A)	0.9800
C(9)-C(14)	1.435(5)	C(30)-H(30B)	0.9800
C(10)-H(10)	0.9500	C(30)-H(30C)	0.9800
C(10)-C(11)	1.360(5)		
C(11)-H(11)	0.9500	C(2)-C(1)-C(14)	120.0(3)
C(11)-C(12)	1.408(5)	C(2)-C(1)-C(15)	120.9(3)
C(12)-H(12)	0.9500	C(14)-C(1)-C(15)	119.1(3)
C(12)-C(13)	1.372(5)	C(1)-C(2)-C(3)	123.3(3)
C(13)-H(13)	0.9500	C(1)-C(2)-C(7)	119.7(3)
C(13)-C(14)	1.428(5)	C(3)-C(2)-C(7)	116.9(3)
C(15)-H(15)	0.9500	C(2)-C(3)-H(3)	119.4
C(15)-C(16)	1.336(5)	C(4)-C(3)-C(2)	121.3(3)
C(16)-H(16)	0.9500	C(4)-C(3)-H(3)	119.4
C(16)-C(17)	1.487(5)	C(3)-C(4)-H(4)	119.4
C(17)-O(18)	1.228(4)	C(3)-C(4)-C(5)	121.2(3)
C(17)-C(19)	1.489(5)	C(5)-C(4)-H(4)	119.4
C(19)-C(20)	1.394(5)	C(4)-C(5)-H(5)	120.1
C(19)-C(24)	1.389(5)	C(6)-C(5)-C(4)	119.8(3)
C(20)-H(20)	0.9500	C(6)-C(5)-H(5)	120.1
C(20)-C(21)	1.396(5)	C(5)-C(6)-H(6)	119.4

C(5)-C(6)-C(7)	121.1(3)	C(24)-C(19)-C(20)	121.5(3)
C(7)-C(6)-H(6)	119.4	C(19)-C(20)-H(20)	120.4
C(6)-C(7)-C(2)	119.4(3)	C(19)-C(20)-C(21)	119.2(3)
C(8)-C(7)-C(2)	119.3(3)	C(21)-C(20)-H(20)	120.4
C(8)-C(7)-C(6)	121.2(3)	C(20)-C(21)-C(22)	119.4(3)
C(7)-C(8)-H(8)	119.2	O(25)-C(21)-C(20)	125.5(3)
C(9)-C(8)-C(7)	121.7(3)	O(25)-C(21)-C(22)	115.1(3)
C(9)-C(8)-H(8)	119.2	C(23)-C(22)-C(21)	120.3(3)
C(8)-C(9)-C(10)	121.8(3)	O(27)-C(22)-C(21)	119.0(3)
C(8)-C(9)-C(14)	119.7(3)	O(27)-C(22)-C(23)	120.6(3)
C(14)-C(9)-C(10)	118.5(3)	C(24)-C(23)-C(22)	120.2(3)
C(9)-C(10)-H(10)	119.2	O(29)-C(23)-C(22)	115.3(3)
C(11)-C(10)-C(9)	121.5(3)	O(29)-C(23)-C(24)	124.5(3)
C(11)-C(10)-H(10)	119.2	C(19)-C(24)-C(23)	119.1(3)
C(10)-C(11)-H(11)	120.1	C(19)-C(24)-H(24)	120.4
C(10)-C(11)-C(12)	119.8(3)	C(23)-C(24)-H(24)	120.4
C(12)-C(11)-H(11)	120.1	C(21)-O(25)-C(26)	117.0(3)
C(11)-C(12)-H(12)	119.5	O(25)-C(26)-H(26A)	109.5
C(13)-C(12)-C(11)	121.1(3)	O(25)-C(26)-H(26B)	109.5
C(13)-C(12)-H(12)	119.5	O(25)-C(26)-H(26C)	109.5
C(12)-C(13)-H(13)	119.5	H(26A)-C(26)-H(26B)	109.5
C(12)-C(13)-C(14)	121.0(3)	H(26A)-C(26)-H(26C)	109.5
C(14)-C(13)-H(13)	119.5	H(26B)-C(26)-H(26C)	109.5
C(1)-C(14)-C(9)	119.4(3)	C(22)-O(27)-C(28)	111.7(3)
C(1)-C(14)-C(13)	122.4(3)	O(27)-C(28)-H(28A)	109.5
C(13)-C(14)-C(9)	118.1(3)	O(27)-C(28)-H(28B)	109.5
C(1)-C(15)-H(15)	117.7	O(27)-C(28)-H(28C)	109.5
C(16)-C(15)-C(1)	124.6(3)	H(28A)-C(28)-H(28B)	109.5
C(16)-C(15)-H(15)	117.7	H(28A)-C(28)-H(28C)	109.5
C(15)-C(16)-H(16)	120.0	H(28B)-C(28)-H(28C)	109.5
C(15)-C(16)-C(17)	120.1(3)	C(23)-O(29)-C(30)	117.3(3)
C(17)-C(16)-H(16)	120.0	O(29)-C(30)-H(30A)	109.5
C(16)-C(17)-C(19)	119.4(3)	O(29)-C(30)-H(30B)	109.5
O(18)-C(17)-C(16)	120.7(3)	O(29)-C(30)-H(30C)	109.5
O(18)-C(17)-C(19)	119.9(3)	H(30A)-C(30)-H(30B)	109.5
C(20)-C(19)-C(17)	121.0(3)	H(30A)-C(30)-H(30C)	109.5
C(24)-C(19)-C(17)	117.2(3)	H(30B)-C(30)-H(30C)	109.5

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd654b. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	16(2)	6(2)	19(2)	3(1)	0(1)	2(1)
C(2)	17(2)	8(2)	15(2)	5(1)	0(1)	1(1)
C(3)	15(2)	13(2)	29(2)	1(2)	1(1)	1(1)
C(4)	19(2)	19(2)	23(2)	1(2)	4(2)	5(2)
C(5)	26(2)	13(2)	21(2)	-2(2)	2(2)	4(2)
C(6)	22(2)	11(2)	26(2)	0(2)	-1(2)	0(2)
C(7)	15(2)	9(2)	23(2)	2(2)	1(1)	1(1)
C(8)	17(2)	13(2)	20(2)	2(2)	1(1)	-2(1)
C(9)	18(2)	10(2)	19(2)	4(2)	2(1)	1(1)
C(10)	15(2)	20(2)	27(2)	1(2)	2(2)	-2(2)
C(11)	16(2)	23(2)	28(2)	3(2)	6(2)	3(2)
C(12)	23(2)	16(2)	17(2)	-2(2)	4(1)	6(2)
C(13)	19(2)	13(2)	21(2)	-2(2)	-1(1)	1(1)
C(14)	18(2)	8(2)	14(2)	1(1)	0(1)	2(1)
C(15)	20(2)	9(2)	21(2)	2(2)	6(1)	-1(1)
C(16)	18(2)	7(2)	25(2)	0(2)	5(1)	-1(1)
C(17)	18(2)	7(2)	23(2)	-1(2)	6(1)	-1(1)
O(18)	18(1)	10(1)	34(2)	0(1)	4(1)	-1(1)
C(19)	14(2)	13(2)	24(2)	-5(2)	5(1)	1(1)
C(20)	14(2)	10(2)	28(2)	-2(2)	4(1)	-1(1)
C(21)	23(2)	11(2)	17(2)	-2(2)	3(1)	2(1)
C(22)	15(2)	18(2)	18(2)	-3(2)	0(1)	3(1)
C(23)	15(2)	14(2)	27(2)	-4(2)	4(1)	-2(2)
C(24)	19(2)	11(2)	23(2)	0(2)	7(1)	1(1)
O(25)	20(1)	14(1)	29(2)	5(1)	-1(1)	-3(1)
C(26)	23(2)	18(2)	26(2)	0(2)	3(2)	-3(2)
O(27)	16(1)	22(1)	30(2)	-2(1)	-2(1)	3(1)
C(28)	26(2)	28(2)	29(2)	-6(2)	-2(2)	0(2)
O(29)	14(1)	22(2)	35(2)	3(1)	2(1)	-4(1)
C(30)	19(2)	14(2)	39(2)	1(2)	7(2)	-2(2)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd654b.

	x	y	z	U(eq)
H(3)	2691	4435	8076	23
H(4)	2640	7512	9289	24
H(5)	3235	10477	9667	24
H(6)	3911	10079	8939	24
H(8)	4380	7852	7789	20
H(10)	4866	5641	6646	25
H(11)	4956	2518	5462	26
H(12)	4341	-226	4919	23
H(13)	3646	109	5583	21
H(15)	3051	92	6584	19
H(16)	2508	4308	6090	20
H(20)	2000	4654	4476	21
H(24)	1372	-1084	5753	21
H(26A)	1569	8790	2429	33
H(26B)	1898	6462	2736	33
H(26C)	1776	8268	3657	33
H(28A)	134	2254	1892	42
H(28B)	446	127	2513	42
H(28C)	683	2199	1873	42
H(30A)	717	-3578	5285	36
H(30B)	202	-2796	5451	36
H(30C)	642	-1566	6170	36

Table 7. Torsion angles [°] for tcd654b.

C(1)-C(2)-C(3)-C(4)	-179.8(3)	C(15)-C(1)-C(2)-C(3)	8.2(5)
C(1)-C(2)-C(7)-C(6)	178.1(3)	C(15)-C(1)-C(2)-C(7)	-175.3(3)
C(1)-C(2)-C(7)-C(8)	-1.4(5)	C(15)-C(1)-C(14)-C(9)	175.6(3)
C(1)-C(15)-C(16)-C(17)	-178.7(3)	C(15)-C(1)-C(14)-C(13)	-1.7(5)
C(2)-C(1)-C(14)-C(9)	-3.7(5)	C(15)-C(16)-C(17)-O(18)	10.3(5)
C(2)-C(1)-C(14)-C(13)	179.0(3)	C(15)-C(16)-C(17)-C(19)	-170.5(3)
C(2)-C(1)-C(15)-C(16)	51.2(5)	C(16)-C(17)-C(19)-C(20)	33.3(5)
C(2)-C(3)-C(4)-C(5)	0.7(6)	C(16)-C(17)-C(19)-C(24)	-152.4(3)
C(2)-C(7)-C(8)-C(9)	-1.5(5)	C(17)-C(19)-C(20)-C(21)	170.3(3)
C(3)-C(2)-C(7)-C(6)	-5.3(5)	C(17)-C(19)-C(24)-C(23)	-173.6(3)
C(3)-C(2)-C(7)-C(8)	175.3(3)	O(18)-C(17)-C(19)-C(20)	-147.5(4)
C(3)-C(4)-C(5)-C(6)	-3.6(6)	O(18)-C(17)-C(19)-C(24)	26.8(5)
C(4)-C(5)-C(6)-C(7)	1.8(5)	C(19)-C(20)-C(21)-C(22)	3.3(5)
C(5)-C(6)-C(7)-C(2)	2.7(5)	C(19)-C(20)-C(21)-O(25)	-177.3(3)
C(5)-C(6)-C(7)-C(8)	-177.9(3)	C(20)-C(19)-C(24)-C(23)	0.6(5)
C(6)-C(7)-C(8)-C(9)	179.1(3)	C(20)-C(21)-C(22)-C(23)	0.2(5)
C(7)-C(2)-C(3)-C(4)	3.6(5)	C(20)-C(21)-C(22)-O(27)	178.4(3)
C(7)-C(8)-C(9)-C(10)	179.8(3)	C(20)-C(21)-O(25)-C(26)	1.1(5)
C(7)-C(8)-C(9)-C(14)	1.7(5)	C(21)-C(22)-C(23)-C(24)	-3.4(5)
C(8)-C(9)-C(10)-C(11)	-178.8(4)	C(21)-C(22)-C(23)-O(29)	177.3(3)
C(8)-C(9)-C(14)-C(1)	0.9(5)	C(21)-C(22)-O(27)-C(28)	96.6(4)
C(8)-C(9)-C(14)-C(13)	178.3(3)	C(22)-C(21)-O(25)-C(26)	-179.5(3)
C(9)-C(10)-C(11)-C(12)	0.9(6)	C(22)-C(23)-C(24)-C(19)	3.0(5)
C(10)-C(9)-C(14)-C(1)	-177.3(3)	C(22)-C(23)-O(29)-C(30)	-177.5(3)
C(10)-C(9)-C(14)-C(13)	0.1(5)	C(23)-C(22)-O(27)-C(28)	-85.2(4)
C(10)-C(11)-C(12)-C(13)	-0.6(6)	C(24)-C(19)-C(20)-C(21)	-3.7(5)
C(11)-C(12)-C(13)-C(14)	0.0(5)	C(24)-C(23)-O(29)-C(30)	3.2(5)
C(12)-C(13)-C(14)-C(1)	177.5(3)	O(25)-C(21)-C(22)-C(23)	-179.3(3)
C(12)-C(13)-C(14)-C(9)	0.2(5)	O(25)-C(21)-C(22)-O(27)	-1.1(5)
C(14)-C(1)-C(2)-C(3)	-172.5(3)	O(27)-C(22)-C(23)-C(24)	178.4(3)
C(14)-C(1)-C(2)-C(7)	4.0(5)	O(27)-C(22)-C(23)-O(29)	-0.9(5)
C(14)-C(1)-C(15)-C(16)	-128.0(4)	O(29)-C(23)-C(24)-C(19)	-177.7(3)
C(14)-C(9)-C(10)-C(11)	-0.7(5)		

Table 8. Hydrogen bonds for tcd654b [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(16)-H(16)...O(18)#1	0.95	2.44	3.382(4)	173.3
C(26)-H(26B)...O(18)#2	0.98	2.56	3.294(5)	131.2

Symmetry transformations used to generate equivalent atoms:

#1 $x,y+1,z$ #2 $x,-y+1/2,z-1/2$



Small Molecule X-ray Facility School Of Chemistry

Trinity College Dublin
The University of Dublin

Structure Report

Filename: TCD1348

Submitted by: James McKeown
Reference: KAHA
Group: Meegan

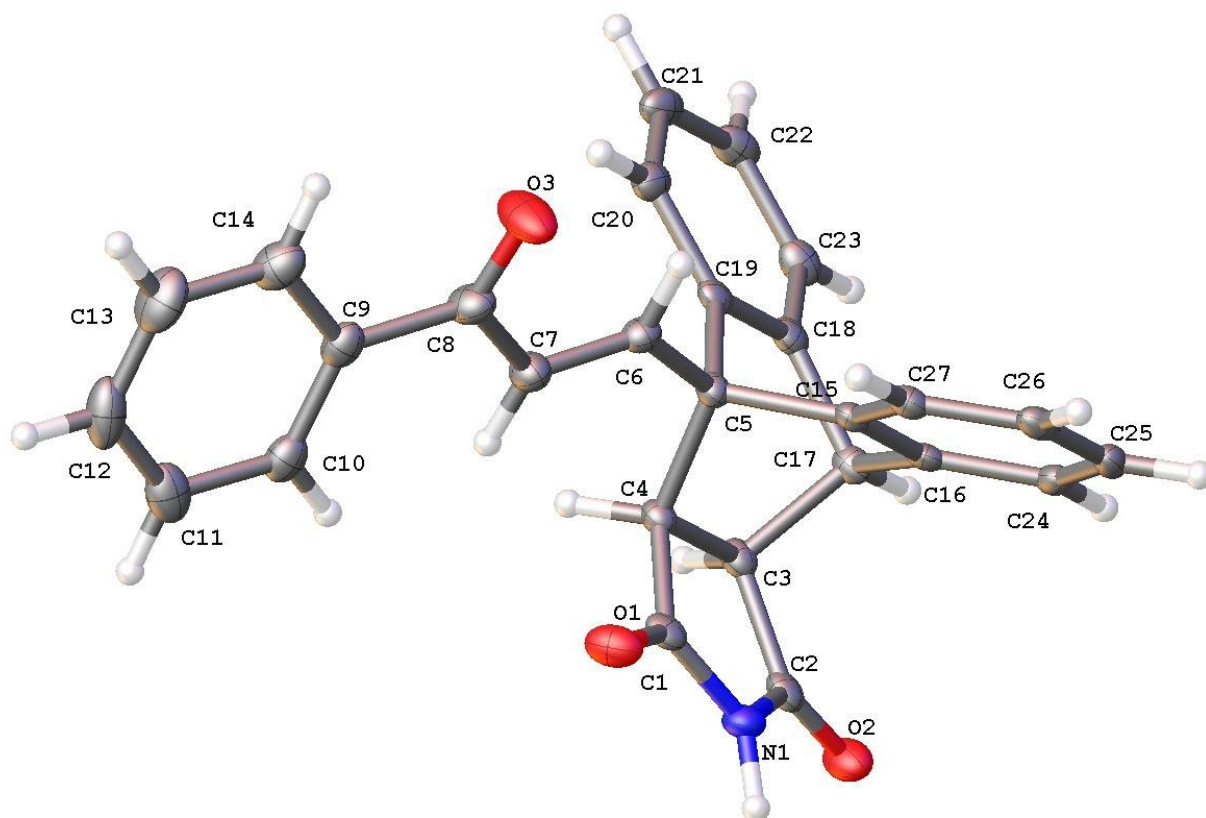


Fig. 1. Molecular structure of TCD1348 with atomic displacement shown at 50% probability.

17/09/19

Author: Brendan Twamley

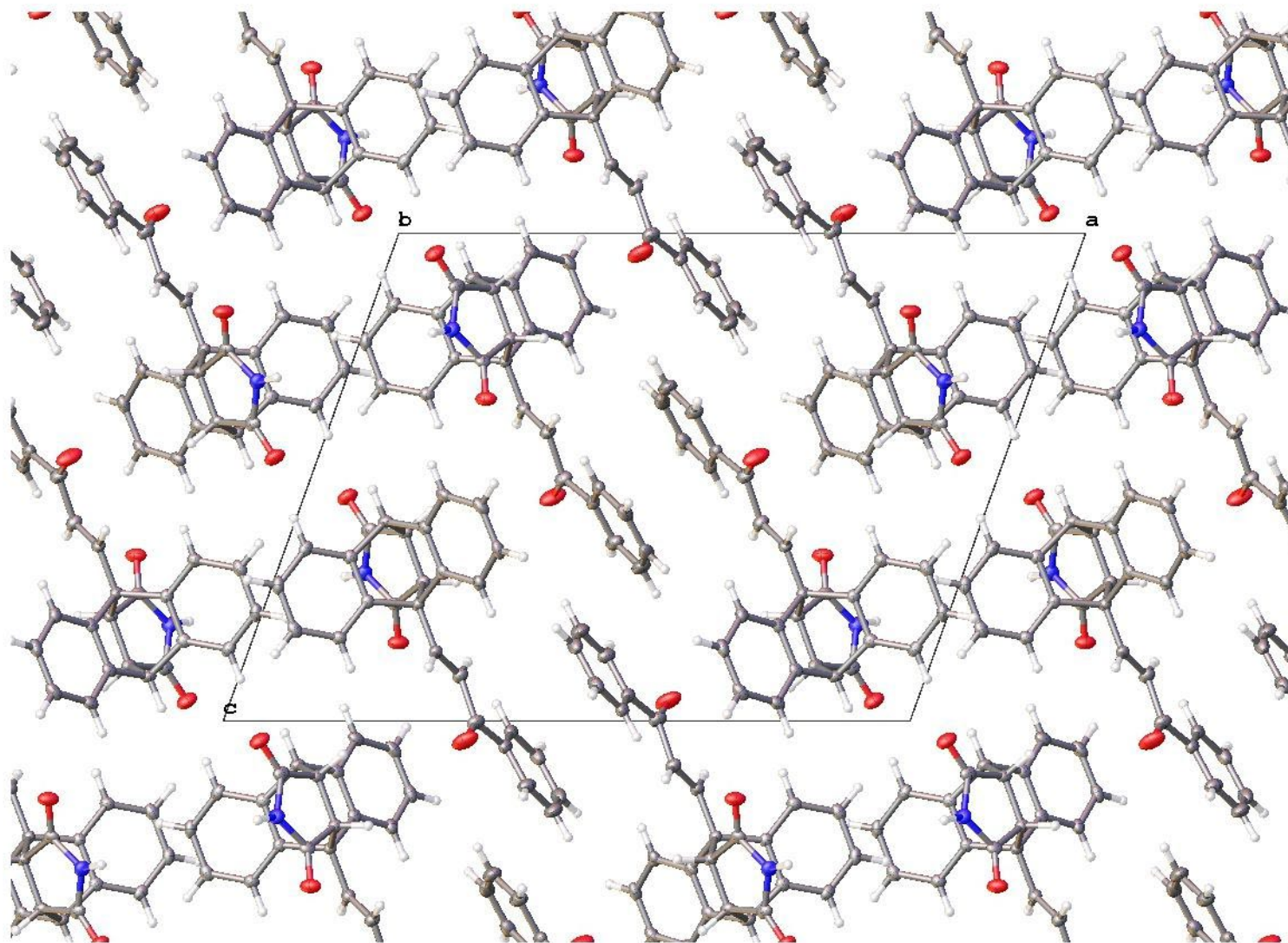


Fig. 2. Packing diagram of TCD1348 viewed normal to the b-axis. Displacement shown at 50% probability.

Crystal Structure Report for TCD1348

A specimen of $\text{C}_{27}\text{H}_{19}\text{NO}_3$, approximate dimensions 0.063 mm x 0.132 mm x 0.220 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured ($\lambda = 0.71073 \text{ \AA}$) at 100(2)K on a Bruker D8 Quest Eco with an Oxford Cryostream low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 339 frames were collected. The total exposure time was 5.65 hours. The integration of the data using a **monoclinic** unit cell yielded a total of 20477 reflections to a maximum θ angle of 27.25° (0.78 $\text{\AA}$ resolution), of which 4272 were independent (average redundancy 4.793, completeness = 99.7%, $R_{\text{int}} = 7.65\%$, $R_{\text{sig}} = 5.33\%$) and 2964 (69.38%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 20.1992(16) \text{ \AA}$, $b = 6.5975(5) \text{ \AA}$, $c = 15.2217(11) \text{ \AA}$, $\beta = 109.811(2)^\circ$, volume = $1908.5(3) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of reflections above $20 \sigma(I)$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.934. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.9800 and 0.9940.

The structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimisation with Olex2, using the space group $P2_1/c$, with $Z = 4$ for the formula unit, $\text{C}_{27}\text{H}_{19}\text{NO}_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 280 variables converged at $R1 = 5.08\%$, for the observed data and $wR2 = 12.23\%$ for all data. The goodness-of-fit was 1.043. The largest peak in the final difference electron density synthesis was $0.234 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.296 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.060 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.411 g/cm^3 and $F(000)$, 848 e^- .

Refinement Note: N-H hydrogen located on the difference map and refined with restraints (DFIX). The molecule has a chiral centre at C3, C4, however it is a racemate and the R configuration is shown (centrosymmetric space group).

References:

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Table 1: Data collection details for TCD1348.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	41.320	27.33	210.08	226.30	54.76	1.50	113	60.00	0.71073	50	20.0	100
Omega	41.320	27.33	210.08	164.09	54.76	1.50	113	60.00	0.71073	50	20.0	100
Omega	41.320	27.33	210.08	75.44	54.76	1.50	113	60.00	0.71073	50	20.0	100

Table 2. Crystal data and structure refinement for tcd1348.

Identification code	tcd1348	
Empirical formula	C ₂₇ H ₁₉ NO ₃	
Formula weight	405.43	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 20.1992(16) Å	α = 90°.
	b = 6.5975(5) Å	β = 109.811(2)°.
	c = 15.2217(11) Å	γ = 90°.
Volume	1908.5(3) Å ³	
Z	4	
Density (calculated)	1.411 Mg/m ³	
Absorption coefficient	0.092 mm ⁻¹	
F(000)	848	
Crystal size	0.22 x 0.132 x 0.063 mm ³	
Theta range for data collection	2.678 to 27.252°.	
Index ranges	-26 ≤ h ≤ 25, -8 ≤ k ≤ 8, -19 ≤ l ≤ 19	
Reflections collected	20477	
Independent reflections	4272 [R(int) = 0.0765]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7455 and 0.6960	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4272 / 1 / 280	
Goodness-of-fit on F ²	1.043	
Final R indices [I > 2σ(I)]	R1 = 0.0508, wR2 = 0.1051	
R indices (all data)	R1 = 0.0872, wR2 = 0.1223	
Largest diff. peak and hole	0.234 and -0.296 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd1348. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	2139(1)	6713(2)	3395(1)	27(1)
O(2)	634(1)	6447(2)	410(1)	29(1)
O(3)	3626(1)	1322(2)	5410(1)	41(1)
N(1)	1280(1)	6774(2)	1966(1)	20(1)
C(1)	1892(1)	6062(3)	2612(1)	18(1)
C(2)	1123(1)	5950(3)	1085(1)	20(1)
C(3)	1663(1)	4336(3)	1137(1)	18(1)
C(4)	2183(1)	4392(3)	2154(1)	16(1)
C(5)	2225(1)	2251(3)	2617(1)	16(1)
C(6)	2736(1)	2049(3)	3591(1)	18(1)
C(7)	3211(1)	3374(3)	4071(1)	22(1)
C(8)	3685(1)	2904(3)	5031(1)	25(1)
C(9)	4240(1)	4400(3)	5523(1)	23(1)
C(10)	4247(1)	6378(3)	5225(1)	23(1)
C(11)	4761(1)	7718(4)	5737(2)	31(1)
C(12)	5277(1)	7072(4)	6544(2)	36(1)
C(13)	5275(1)	5111(4)	6839(2)	38(1)
C(14)	4764(1)	3776(4)	6339(1)	30(1)
C(15)	1475(1)	1721(3)	2554(1)	15(1)
C(16)	995(1)	1704(3)	1642(1)	16(1)
C(17)	1318(1)	2206(3)	908(1)	18(1)
C(18)	1925(1)	774(3)	1044(1)	18(1)
C(19)	2413(1)	799(3)	1952(1)	17(1)
C(20)	3004(1)	-417(3)	2174(1)	20(1)
C(21)	3106(1)	-1650(3)	1490(2)	24(1)
C(22)	2624(1)	-1674(3)	597(1)	24(1)
C(23)	2030(1)	-453(3)	369(1)	22(1)
C(24)	301(1)	1166(3)	1474(1)	19(1)
C(25)	90(1)	625(3)	2221(1)	20(1)
C(26)	559(1)	663(3)	3125(1)	20(1)
C(27)	1254(1)	1230(3)	3298(1)	18(1)

Table 4. Bond lengths [Å] and angles [°] for *tcd1348*.

O(1)-C(1)	1.205(2)	C(17)-H(17)	1.0000
O(2)-C(2)	1.205(2)	C(17)-C(18)	1.507(3)
O(3)-C(8)	1.219(3)	C(18)-C(19)	1.399(3)
N(1)-H(1)	0.9686	C(18)-C(23)	1.380(3)
N(1)-C(1)	1.375(2)	C(19)-C(20)	1.382(3)
N(1)-C(2)	1.380(2)	C(20)-H(20)	0.9500
C(1)-C(4)	1.524(3)	C(20)-C(21)	1.390(3)
C(2)-C(3)	1.508(3)	C(21)-H(21)	0.9500
C(3)-H(3)	1.0000	C(21)-C(22)	1.379(3)
C(3)-C(4)	1.548(2)	C(22)-H(22)	0.9500
C(3)-C(17)	1.555(3)	C(22)-C(23)	1.387(3)
C(4)-H(4)	1.0000	C(23)-H(23)	0.9500
C(4)-C(5)	1.568(2)	C(24)-H(24)	0.9500
C(5)-C(6)	1.498(3)	C(24)-C(25)	1.389(3)
C(5)-C(15)	1.526(3)	C(25)-H(25)	0.9500
C(5)-C(19)	1.532(3)	C(25)-C(26)	1.381(3)
C(6)-H(6)	0.9500	C(26)-H(26)	0.9500
C(6)-C(7)	1.321(3)	C(26)-C(27)	1.388(3)
C(7)-H(7)	0.9500	C(27)-H(27)	0.9500
C(7)-C(8)	1.483(3)		
C(8)-C(9)	1.491(3)	C(1)-N(1)-H(1)	125.6
C(9)-C(10)	1.384(3)	C(1)-N(1)-C(2)	114.51(16)
C(9)-C(14)	1.393(3)	C(2)-N(1)-H(1)	118.5
C(10)-H(10)	0.9500	O(1)-C(1)-N(1)	123.80(18)
C(10)-C(11)	1.385(3)	O(1)-C(1)-C(4)	127.98(18)
C(11)-H(11)	0.9500	N(1)-C(1)-C(4)	108.21(15)
C(11)-C(12)	1.381(3)	O(2)-C(2)-N(1)	125.04(19)
C(12)-H(12)	0.9500	O(2)-C(2)-C(3)	127.31(18)
C(12)-C(13)	1.370(4)	N(1)-C(2)-C(3)	107.66(16)
C(13)-H(13)	0.9500	C(2)-C(3)-H(3)	109.9
C(13)-C(14)	1.374(3)	C(2)-C(3)-C(4)	105.61(15)
C(14)-H(14)	0.9500	C(2)-C(3)-C(17)	111.34(15)
C(15)-C(16)	1.398(3)	C(4)-C(3)-H(3)	109.9
C(15)-C(27)	1.388(3)	C(4)-C(3)-C(17)	110.24(15)
C(16)-C(17)	1.508(3)	C(17)-C(3)-H(3)	109.9
C(16)-C(24)	1.384(3)	C(1)-C(4)-C(3)	103.79(15)

C(1)-C(4)-H(4)	109.4	C(16)-C(15)-C(5)	113.75(16)
C(1)-C(4)-C(5)	114.58(15)	C(27)-C(15)-C(5)	126.13(17)
C(3)-C(4)-H(4)	109.4	C(27)-C(15)-C(16)	120.09(17)
C(3)-C(4)-C(5)	110.06(14)	C(15)-C(16)-C(17)	113.97(16)
C(5)-C(4)-H(4)	109.4	C(24)-C(16)-C(15)	120.21(17)
C(6)-C(5)-C(4)	116.07(15)	C(24)-C(16)-C(17)	125.75(17)
C(6)-C(5)-C(15)	112.17(15)	C(3)-C(17)-H(17)	112.2
C(6)-C(5)-C(19)	110.90(15)	C(16)-C(17)-C(3)	107.86(15)
C(15)-C(5)-C(4)	106.07(15)	C(16)-C(17)-H(17)	112.2
C(15)-C(5)-C(19)	106.19(14)	C(18)-C(17)-C(3)	104.63(15)
C(19)-C(5)-C(4)	104.72(14)	C(18)-C(17)-C(16)	107.31(15)
C(5)-C(6)-H(6)	116.0	C(18)-C(17)-H(17)	112.2
C(7)-C(6)-C(5)	128.06(18)	C(19)-C(18)-C(17)	113.30(16)
C(7)-C(6)-H(6)	116.0	C(23)-C(18)-C(17)	126.13(17)
C(6)-C(7)-H(7)	119.6	C(23)-C(18)-C(19)	120.56(18)
C(6)-C(7)-C(8)	120.82(19)	C(18)-C(19)-C(5)	114.27(16)
C(8)-C(7)-H(7)	119.6	C(20)-C(19)-C(5)	126.04(17)
O(3)-C(8)-C(7)	120.77(19)	C(20)-C(19)-C(18)	119.68(17)
O(3)-C(8)-C(9)	120.48(19)	C(19)-C(20)-H(20)	120.3
C(7)-C(8)-C(9)	118.76(18)	C(19)-C(20)-C(21)	119.46(18)
C(10)-C(9)-C(8)	123.33(18)	C(21)-C(20)-H(20)	120.3
C(10)-C(9)-C(14)	118.8(2)	C(20)-C(21)-H(21)	119.7
C(14)-C(9)-C(8)	117.84(19)	C(22)-C(21)-C(20)	120.67(19)
C(9)-C(10)-H(10)	119.8	C(22)-C(21)-H(21)	119.7
C(9)-C(10)-C(11)	120.5(2)	C(21)-C(22)-H(22)	119.9
C(11)-C(10)-H(10)	119.8	C(21)-C(22)-C(23)	120.17(18)
C(10)-C(11)-H(11)	120.0	C(23)-C(22)-H(22)	119.9
C(12)-C(11)-C(10)	119.9(2)	C(18)-C(23)-C(22)	119.46(18)
C(12)-C(11)-H(11)	120.0	C(18)-C(23)-H(23)	120.3
C(11)-C(12)-H(12)	120.1	C(22)-C(23)-H(23)	120.3
C(13)-C(12)-C(11)	119.9(2)	C(16)-C(24)-H(24)	120.4
C(13)-C(12)-H(12)	120.1	C(16)-C(24)-C(25)	119.16(18)
C(12)-C(13)-H(13)	119.7	C(25)-C(24)-H(24)	120.4
C(12)-C(13)-C(14)	120.6(2)	C(24)-C(25)-H(25)	119.6
C(14)-C(13)-H(13)	119.7	C(26)-C(25)-C(24)	120.88(18)
C(9)-C(14)-H(14)	119.8	C(26)-C(25)-H(25)	119.6
C(13)-C(14)-C(9)	120.4(2)	C(25)-C(26)-H(26)	119.9
C(13)-C(14)-H(14)	119.8	C(25)-C(26)-C(27)	120.13(18)

C(27)-C(26)-H(26)	119.9	C(15)-C(27)-H(27)	120.3
C(15)-C(27)-C(26)	119.49(17)	C(26)-C(27)-H(27)	120.3

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd1348. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	39(1)	22(1)	18(1)	-1(1)	6(1)	4(1)
O(2)	29(1)	23(1)	26(1)	4(1)	-2(1)	1(1)
O(3)	48(1)	28(1)	32(1)	10(1)	-7(1)	-6(1)
N(1)	23(1)	14(1)	23(1)	3(1)	6(1)	3(1)
C(1)	25(1)	13(1)	17(1)	3(1)	7(1)	-2(1)
C(2)	23(1)	16(1)	21(1)	3(1)	5(1)	-6(1)
C(3)	20(1)	16(1)	15(1)	2(1)	5(1)	-4(1)
C(4)	18(1)	14(1)	16(1)	2(1)	4(1)	-2(1)
C(5)	16(1)	14(1)	16(1)	1(1)	4(1)	-1(1)
C(6)	18(1)	18(1)	17(1)	3(1)	5(1)	3(1)
C(7)	21(1)	20(1)	22(1)	2(1)	3(1)	1(1)
C(8)	25(1)	23(1)	22(1)	2(1)	2(1)	4(1)
C(9)	19(1)	30(1)	20(1)	-4(1)	6(1)	2(1)
C(10)	19(1)	32(1)	20(1)	-3(1)	8(1)	2(1)
C(11)	32(1)	37(1)	29(1)	-8(1)	18(1)	-9(1)
C(12)	26(1)	57(2)	28(1)	-15(1)	12(1)	-15(1)
C(13)	28(1)	58(2)	22(1)	-4(1)	1(1)	0(1)
C(14)	28(1)	36(1)	21(1)	-3(1)	4(1)	6(1)
C(15)	17(1)	10(1)	20(1)	0(1)	6(1)	0(1)
C(16)	21(1)	9(1)	20(1)	0(1)	7(1)	1(1)
C(17)	21(1)	17(1)	14(1)	0(1)	3(1)	-2(1)
C(18)	22(1)	14(1)	20(1)	1(1)	10(1)	-4(1)
C(19)	20(1)	12(1)	21(1)	2(1)	10(1)	-3(1)
C(20)	20(1)	19(1)	24(1)	4(1)	10(1)	0(1)
C(21)	24(1)	20(1)	34(1)	4(1)	19(1)	3(1)
C(22)	34(1)	18(1)	28(1)	-2(1)	21(1)	-1(1)
C(23)	28(1)	21(1)	19(1)	-1(1)	12(1)	-6(1)
C(24)	17(1)	12(1)	24(1)	-2(1)	3(1)	2(1)
C(25)	16(1)	13(1)	33(1)	-1(1)	11(1)	-1(1)
C(26)	26(1)	12(1)	27(1)	0(1)	15(1)	1(1)
C(27)	23(1)	12(1)	18(1)	0(1)	7(1)	0(1)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd1348.

	x	y	z	U(eq)
H(1)	1021	7959	2041	24
H(3)	1918	4665	694	21
H(4)	2660	4791	2153	20
H(6)	2715	814	3902	21
H(7)	3250	4644	3798	26
H(10)	3897	6821	4666	28
H(11)	4759	9079	5533	37
H(12)	5632	7986	6894	43
H(13)	5630	4670	7394	45
H(14)	4768	2420	6550	36
H(17)	966	2134	262	22
H(20)	3338	-411	2789	24
H(21)	3513	-2485	1640	28
H(22)	2698	-2528	136	29
H(23)	1699	-461	-248	26
H(24)	-28	1168	856	23
H(25)	-383	223	2109	24
H(26)	407	300	3629	24
H(27)	1575	1282	3920	21

Table 7. Torsion angles [°] for tcd1348.

O(1)-C(1)-C(4)-C(3)	-176.51(19)	C(5)-C(15)-C(27)-C(26)	-176.13(17)
O(1)-C(1)-C(4)-C(5)	63.4(3)	C(5)-C(19)-C(20)-C(21)	-179.47(17)
O(2)-C(2)-C(3)-C(4)	177.78(19)	C(6)-C(5)-C(15)-C(16)	-174.20(16)
O(2)-C(2)-C(3)-C(17)	-62.6(3)	C(6)-C(5)-C(15)-C(27)	4.0(3)
O(3)-C(8)-C(9)-C(10)	-166.0(2)	C(6)-C(5)-C(19)-C(18)	174.84(16)
O(3)-C(8)-C(9)-C(14)	12.0(3)	C(6)-C(5)-C(19)-C(20)	-5.7(2)
N(1)-C(1)-C(4)-C(3)	2.64(19)	C(6)-C(7)-C(8)-O(3)	-3.1(3)
N(1)-C(1)-C(4)-C(5)	-117.41(17)	C(6)-C(7)-C(8)-C(9)	176.50(19)
N(1)-C(2)-C(3)-C(4)	-2.81(19)	C(7)-C(8)-C(9)-C(10)	14.4(3)
N(1)-C(2)-C(3)-C(17)	116.85(16)	C(7)-C(8)-C(9)-C(14)	-167.56(19)
C(1)-N(1)-C(2)-O(2)	-175.68(19)	C(8)-C(9)-C(10)-C(11)	177.18(19)
C(1)-N(1)-C(2)-C(3)	4.9(2)	C(8)-C(9)-C(14)-C(13)	-177.7(2)
C(1)-C(4)-C(5)-C(6)	-66.3(2)	C(9)-C(10)-C(11)-C(12)	0.8(3)
C(1)-C(4)-C(5)-C(15)	59.01(19)	C(10)-C(9)-C(14)-C(13)	0.4(3)
C(1)-C(4)-C(5)-C(19)	171.07(15)	C(10)-C(11)-C(12)-C(13)	-0.4(3)
C(2)-N(1)-C(1)-O(1)	174.35(18)	C(11)-C(12)-C(13)-C(14)	0.0(4)
C(2)-N(1)-C(1)-C(4)	-4.8(2)	C(12)-C(13)-C(14)-C(9)	0.0(3)
C(2)-C(3)-C(4)-C(1)	0.11(18)	C(14)-C(9)-C(10)-C(11)	-0.8(3)
C(2)-C(3)-C(4)-C(5)	123.18(16)	C(15)-C(5)-C(6)-C(7)	-130.5(2)
C(2)-C(3)-C(17)-C(16)	-62.85(19)	C(15)-C(5)-C(19)-C(18)	52.73(19)
C(2)-C(3)-C(17)-C(18)	-176.87(15)	C(15)-C(5)-C(19)-C(20)	-127.76(19)
C(3)-C(4)-C(5)-C(6)	177.19(15)	C(15)-C(16)-C(17)-C(3)	-58.0(2)
C(3)-C(4)-C(5)-C(15)	-57.49(18)	C(15)-C(16)-C(17)-C(18)	54.3(2)
C(3)-C(4)-C(5)-C(19)	54.57(18)	C(15)-C(16)-C(24)-C(25)	-0.7(3)
C(3)-C(17)-C(18)-C(19)	60.05(19)	C(16)-C(15)-C(27)-C(26)	2.0(3)
C(3)-C(17)-C(18)-C(23)	-118.9(2)	C(16)-C(17)-C(18)-C(19)	-54.4(2)
C(4)-C(3)-C(17)-C(16)	54.02(19)	C(16)-C(17)-C(18)-C(23)	126.74(19)
C(4)-C(3)-C(17)-C(18)	-60.00(19)	C(16)-C(24)-C(25)-C(26)	1.5(3)
C(4)-C(5)-C(6)-C(7)	-8.3(3)	C(17)-C(3)-C(4)-C(1)	-120.27(16)
C(4)-C(5)-C(15)-C(16)	58.13(19)	C(17)-C(3)-C(4)-C(5)	2.8(2)
C(4)-C(5)-C(15)-C(27)	-123.66(19)	C(17)-C(16)-C(24)-C(25)	176.08(18)
C(4)-C(5)-C(19)-C(18)	-59.25(19)	C(17)-C(18)-C(19)-C(5)	0.5(2)
C(4)-C(5)-C(19)-C(20)	120.26(19)	C(17)-C(18)-C(19)-C(20)	-179.00(16)
C(5)-C(6)-C(7)-C(8)	-179.01(18)	C(17)-C(18)-C(23)-C(22)	179.04(18)
C(5)-C(15)-C(16)-C(17)	0.2(2)	C(18)-C(19)-C(20)-C(21)	0.0(3)
C(5)-C(15)-C(16)-C(24)	177.31(16)	C(19)-C(5)-C(6)-C(7)	111.0(2)

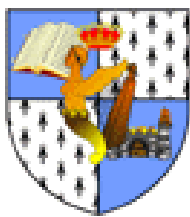
C(19)-C(5)-C(15)-C(16)	-52.9(2)	C(23)-C(18)-C(19)-C(20)	0.0(3)
C(19)-C(5)-C(15)-C(27)	125.31(19)	C(24)-C(16)-C(17)-C(3)	125.07(19)
C(19)-C(18)-C(23)-C(22)	0.2(3)	C(24)-C(16)-C(17)-C(18)	-122.70(19)
C(19)-C(20)-C(21)-C(22)	-0.2(3)	C(24)-C(25)-C(26)-C(27)	-0.6(3)
C(20)-C(21)-C(22)-C(23)	0.3(3)	C(25)-C(26)-C(27)-C(15)	-1.2(3)
C(21)-C(22)-C(23)-C(18)	-0.4(3)	C(27)-C(15)-C(16)-C(17)	-178.18(16)
C(23)-C(18)-C(19)-C(5)	179.51(16)	C(27)-C(15)-C(16)-C(24)	-1.0(3)

Table 8. Hydrogen bonds for tcd1348 [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(22)-H(22)...O(1)#1	0.95	2.56	3.158(2)	121
C(24)-H(24)...O(2)#2	0.95	2.47	3.255(2)	140

Symmetry transformations used to generate equivalent atoms:

#1 $x, -y+1/2, z-1/2$ #2 $-x, -y+1, -z$



Small Molecule X-ray Facility School Of Chemistry



Structure Report

Filename: TCD814b

Submitted by: James Mc Keown

Reference: CC55

Group: Meegan

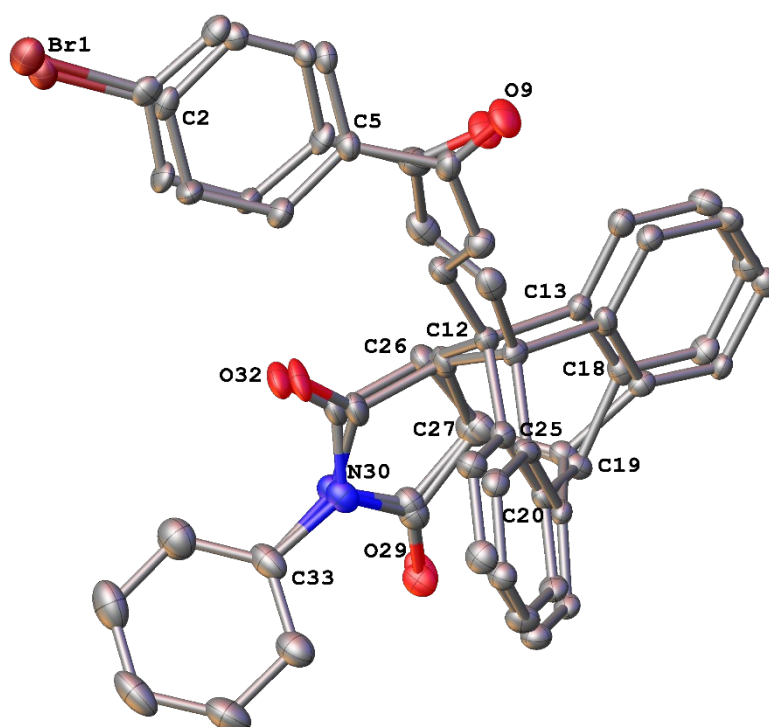
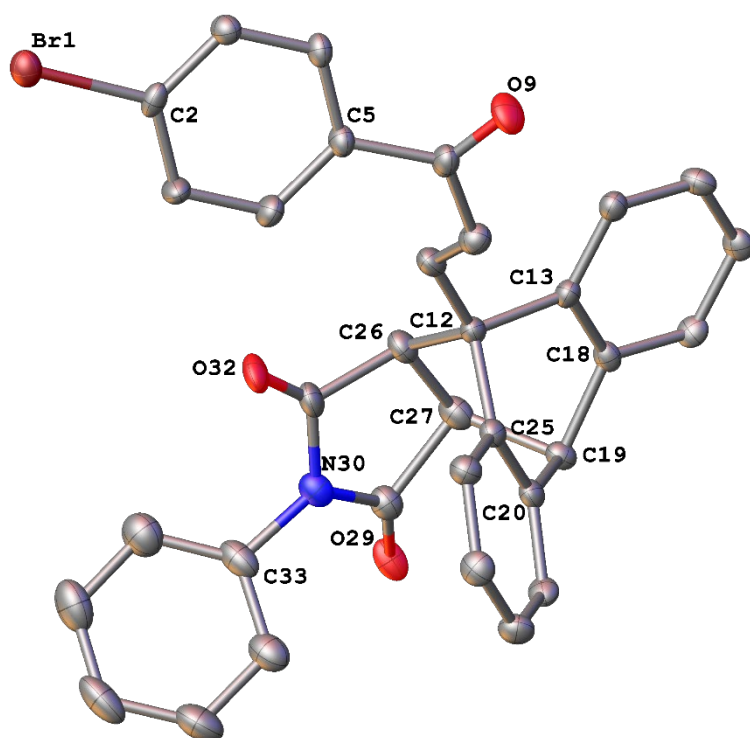


Fig. 1. Disordered asymmetric unit of TCD814b with partial atom labelling for clarity. Moiety occupancy 61:39%. Atomic displacement shown at 50% probability. Hydrogen atoms omitted for clarity.

6/6/17

Author: Brendan Twamley

A



B

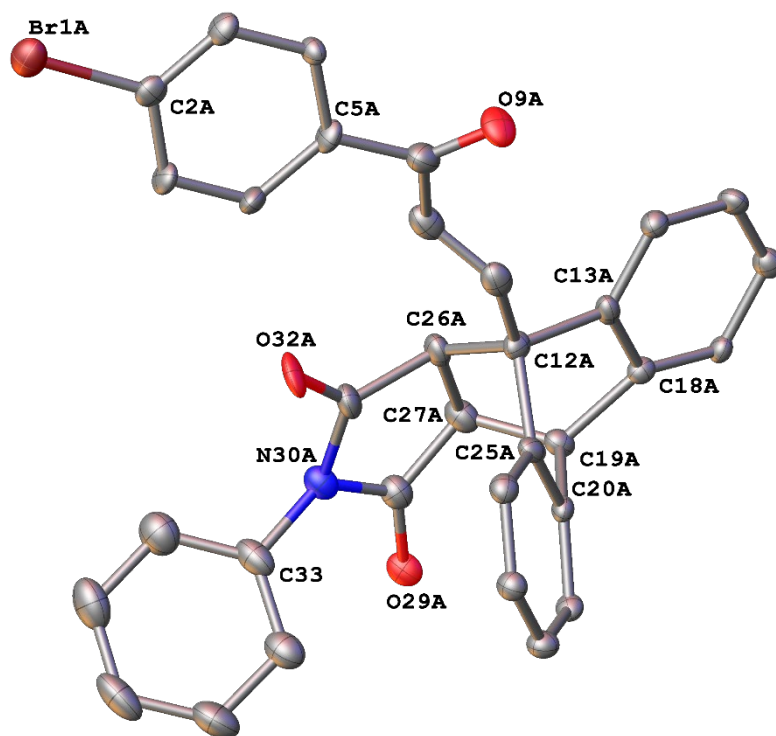


Fig. 2. Individual disordered moieties of TCD814b (A) 61% occupied and (B) 39% occupied. Atomic displacement shown at 50% probability. Hydrogen atoms omitted for clarity.

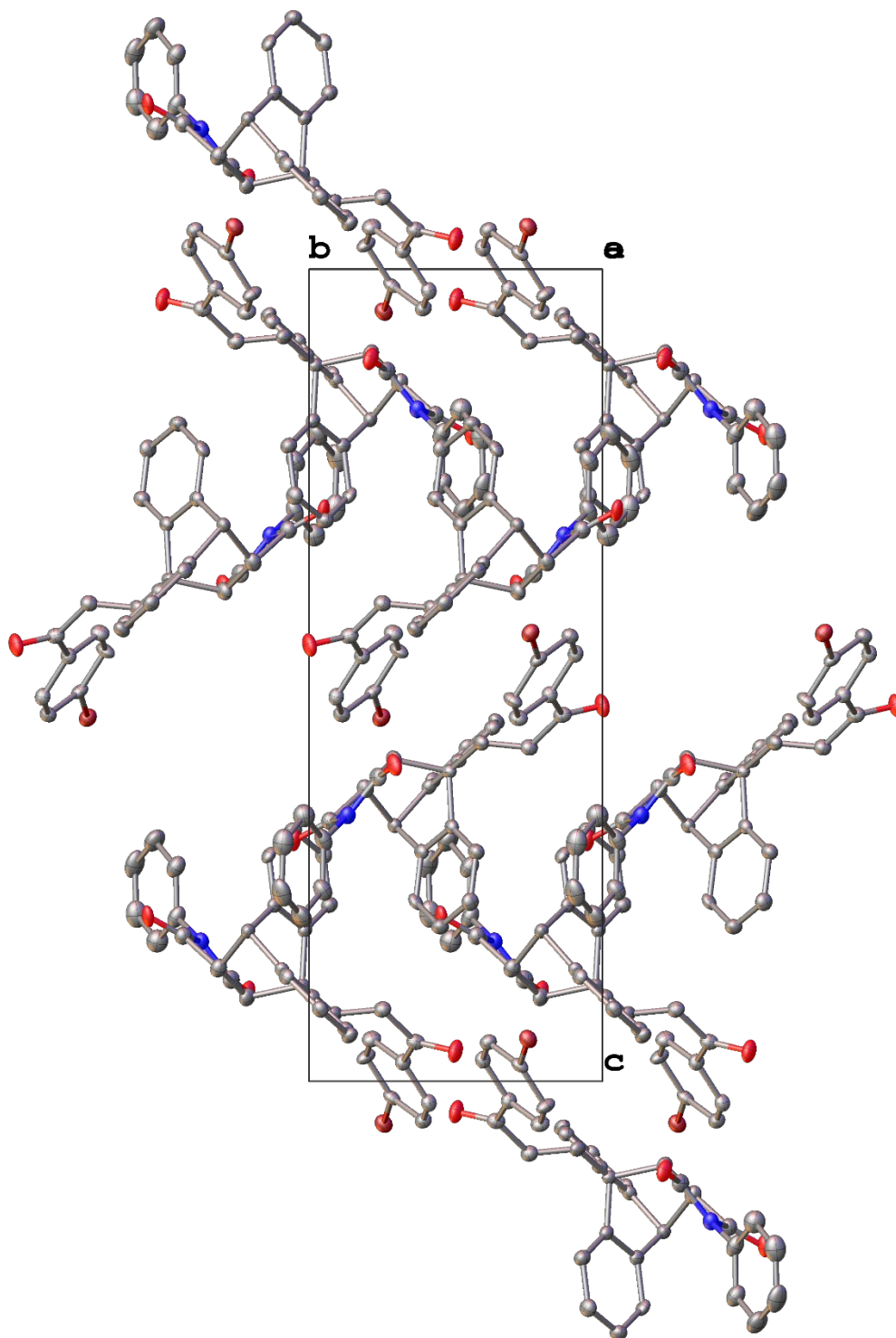


Fig. 3. Packing diagram of the major disordered moiety of TCD814b showing the sinusoidal packing nature of the molecules as viewed normal to the a-axis. Atomic displacement shown at 50% probability. Hydrogen atoms omitted for clarity.

Crystal Structure Report for TCD814b

A clear colourless block fragment-like specimen of $\text{C}_{33}\text{H}_{22}\text{BrNO}_3$, approximate dimensions 0.120 mm x 0.150 mm x 0.230 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K with an Oxford Cryosystems low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 1684 frames were collected. The total exposure time was 7.77 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 65657 reflections to a maximum θ angle of 26.14° (0.81 Å resolution), of which 4872 were independent (average redundancy 13.476, completeness = 99.7%, $R_{\text{int}} = 8.60\%$, $R_{\text{sig}} = 3.69\%$) and 3687 (75.68%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 13.5908(4)$ Å, $b = 8.0761(2)$ Å, $c = 23.2409(6)$ Å, $\beta = 106.0230(10)^\circ$, volume = $2451.84(11)$ Å³, are based upon the refinement of the XYZ-centroids of 9920 reflections above $20\sigma(I)$ with $5.363^\circ < 2\theta < 52.04^\circ$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.908. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6766 and 0.7453.

The structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimisation with Olex2, using the space group $P2_1/c$, with $Z = 4$ for the formula unit, $\text{C}_{33}\text{H}_{22}\text{BrNO}_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 566 variables converged at $R1 = 3.88\%$, for the observed data and $wR2 = 8.32\%$ for all data. The goodness-of-fit was 1.063. The largest peak in the final difference electron density synthesis was $0.351 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.453 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.062 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.518 g/cm^3 and $F(000)$, 1144 e^- .

Refinement Note: The majority of the molecule was disordered in two positions with approx. 61:39% occupancy. Restraints (SIMU, ISOR) and some constraints (EADP) were used in the model for L.S. convergence.

References: see CIF.

Table 1: Data collection details for TCD814b.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Phi	50.517	8.00	0.00	0.00	54.76	1.00	180	5.00	0.71073	50	20.0	100
Phi	50.517	29.14	208.14	0.00	54.76	1.20	300	18.00	0.71073	50	20.0	100
Omega	50.517	-19.41	159.82	0.00	54.76	1.20	151	18.00	0.71073	50	20.0	100
Omega	50.517	-19.41	159.82	160.00	54.76	1.20	151	18.00	0.71073	50	20.0	100
Omega	50.517	-19.41	159.82	80.00	54.76	1.20	151	18.00	0.71073	50	20.0	100
Phi	50.517	29.14	208.14	0.00	54.76	1.20	300	18.00	0.71073	50	20.0	100
Phi	50.517	29.14	30.26	0.00	54.76	1.20	300	18.00	0.71073	50	20.0	100
Omega	50.517	-19.41	159.82	40.00	54.76	1.20	151	18.00	0.71073	50	20.0	100

Crystal Data for $C_{33}H_{22}BrNO_3$ ($M = 560.42$ g/mol): monoclinic, space group $P2_1/c$ (no. 14), $a = 13.5908(4)$ Å, $b = 8.0761(2)$ Å, $c = 23.2409(6)$ Å, $\beta = 106.0230(10)^\circ$, $V = 2451.83(11)$ Å³, $Z = 4$, $T = 100(2)$ K, $\mu(\text{MoK}\alpha) = 1.715$ mm⁻¹, $D_{\text{calc}} = 1.518$ g/cm³, 65657 reflections measured ($5.93^\circ \leq 2\theta \leq 52.282^\circ$), 4872 unique ($R_{\text{int}} = 0.0860$, $R_{\text{sigma}} = 0.0369$) which were used in all calculations. The final R_1 was 0.0388 ($I > 2\sigma(I)$) and wR_2 was 0.0832 (all data).

Table 2. Crystal data and structure refinement for tcd814b.

Identification code	tcd814b	
Empirical formula	C ₃₃ H ₂₂ BrNO ₃	
Formula weight	560.42	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 13.5908(4) Å	α = 90°.
	b = 8.0761(2) Å	β = 106.0230(10)°.
	c = 23.2409(6) Å	γ = 90°.
Volume	2451.83(11) Å ³	
Z	4	
Density (calculated)	1.518 Mg/m ³	
Absorption coefficient	1.715 mm ⁻¹	
F(000)	1144	
Crystal size	0.23 x 0.15 x 0.12 mm ³	
Theta range for data collection	2.965 to 26.141°.	
Index ranges	-16 ≤ h ≤ 16, -10 ≤ k ≤ 10, -28 ≤ l ≤ 28	
Reflections collected	65657	
Independent reflections	4872 [R(int) = 0.0860]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7453 and 0.6766	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4872 / 1044 / 566	
Goodness-of-fit on F ²	1.063	
Final R indices [I > 2σ(I)]	R1 = 0.0388, wR2 = 0.0758	
R indices (all data)	R1 = 0.0622, wR2 = 0.0832	
Largest diff. peak and hole	0.351 and -0.453 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd814b. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
Br(1)	10472(2)	2461(4)	4479(1)	24(1)
Br(1A)	10626(4)	2284(7)	4389(2)	30(1)
O(9A)	6387(12)	713(13)	5347(7)	29(2)
O(29A)	6140(20)	10310(40)	7122(11)	21(3)
O(32A)	7740(20)	6660(20)	6173(12)	26(3)
N(30A)	7120(20)	8960(30)	6644(11)	23(2)
C(2A)	9513(11)	2130(20)	4712(6)	21(2)
C(3A)	9473(10)	3127(19)	5181(5)	20(2)
C(4A)	8615(11)	3123(15)	5387(5)	21(2)
C(5A)	7823(11)	2008(18)	5146(6)	20(2)
C(6A)	7923(11)	943(19)	4697(7)	16(2)
C(7A)	8768(12)	1030(20)	4479(7)	21(2)
C(8A)	6903(12)	1976(17)	5360(6)	26(3)
C(10A)	6586(6)	3565(12)	5609(4)	26(2)
C(11A)	6165(6)	3523(12)	6034(4)	23(2)
C(12A)	5744(9)	4943(14)	6326(5)	18(2)
C(13A)	4561(7)	4778(15)	6180(4)	18(2)
C(14A)	3971(8)	3649(13)	5778(4)	18(2)
C(15A)	2918(8)	3706(17)	5684(5)	19(2)
C(16A)	2478(9)	4717(18)	5998(5)	20(2)
C(17A)	3065(9)	5855(17)	6403(5)	15(2)
C(18A)	4088(9)	5884(15)	6478(5)	16(2)
C(19A)	4940(20)	7140(40)	6755(10)	18(2)
C(20A)	5721(9)	6163(17)	7290(5)	15(2)
C(21A)	6100(10)	6530(18)	7894(5)	16(2)
C(22A)	6981(10)	5702(19)	8201(5)	20(2)
C(23A)	7450(8)	4564(17)	7921(5)	19(2)
C(24A)	7087(8)	4209(15)	7323(5)	19(2)
C(25A)	6194(8)	5008(15)	7007(4)	18(2)
C(26A)	5904(17)	6770(20)	6105(9)	21(2)
C(27A)	5420(40)	8230(50)	6390(30)	24(2)
C(28A)	6210(40)	9220(70)	6733(16)	24(2)
C(31A)	7003(18)	7300(20)	6314(10)	24(2)

C(33)	8105(2)	9513(4)	7024(1)	29(1)
C(34)	8333(2)	9536(4)	7642(1)	32(1)
C(35)	9241(2)	10262(4)	7966(2)	39(1)
C(36)	9909(2)	10939(4)	7675(2)	42(1)
C(37)	9672(2)	10883(4)	7060(2)	41(1)
C(38)	8764(2)	10174(4)	6729(2)	37(1)
O(9)	6363(7)	13(7)	5373(4)	30(2)
O(29)	5990(30)	10490(60)	7033(19)	24(5)
O(32)	7800(13)	7098(13)	6103(7)	26(2)
N(30)	7163(14)	8683(16)	6723(7)	23(2)
C(4)	7707(5)	921(11)	4736(4)	19(2)
C(3)	8555(6)	1109(13)	4518(3)	20(2)
C(2)	9340(5)	2178(13)	4801(3)	19(2)
C(7)	9277(5)	3060(10)	5304(3)	23(2)
C(6)	8429(5)	2873(8)	5522(2)	23(2)
C(5)	7644(5)	1803(9)	5238(3)	19(2)
C(8)	6778(7)	1370(13)	5487(4)	21(2)
C(10)	6447(3)	2505(7)	5887(2)	22(1)
C(11)	6384(4)	4149(8)	5829(3)	21(1)
C(12)	5877(5)	5238(9)	6181(3)	17(1)
C(18)	4241(4)	5914(8)	6372(2)	18(2)
C(17)	3192(4)	5772(9)	6291(3)	19(2)
C(16)	2629(3)	4623(9)	5887(3)	20(2)
C(15)	3115(4)	3615(7)	5564(2)	21(2)
C(14)	4164(4)	3757(7)	5646(2)	20(1)
C(13)	4727(3)	4907(7)	6050(2)	17(1)
C(19)	4876(13)	7080(30)	6851(6)	18(2)
C(23)	7594(4)	4509(9)	7788(2)	23(2)
C(22)	7095(5)	5540(10)	8095(2)	23(2)
C(21)	6210(5)	6371(9)	7787(2)	20(2)
C(20)	5825(4)	6169(9)	7172(2)	18(2)
C(25)	6325(4)	5138(8)	6865(2)	18(1)
C(24)	7209(4)	4308(7)	7173(2)	22(2)
C(26)	5976(11)	7108(12)	6037(6)	23(2)
C(27)	5370(30)	8090(30)	6395(18)	24(2)
C(28)	6200(30)	9300(40)	6793(10)	24(2)
C(31)	7102(10)	7654(13)	6253(6)	24(2)

Table 4. Bond lengths [Å] and angles [°] for tcd814b.

Br(1)-C(2)	1.902(6)	C(17A)-C(18A)	1.352(13)
Br(1A)-C(2A)	1.870(14)	C(18A)-C(19A)	1.54(3)
O(9A)-C(8A)	1.233(14)	C(19A)-H(19A)	1.0000
O(29A)-C(28A)	1.28(5)	C(19A)-C(20A)	1.60(3)
O(32A)-C(31A)	1.25(4)	C(19A)-C(27A)	1.49(6)
N(30A)-C(28A)	1.32(6)	C(20A)-C(21A)	1.387(13)
N(30A)-C(31A)	1.53(3)	C(20A)-C(25A)	1.398(13)
N(30A)-C(33)	1.45(3)	C(21A)-H(21A)	0.9500
C(2A)-C(3A)	1.368(14)	C(21A)-C(22A)	1.385(12)
C(2A)-C(7A)	1.343(13)	C(22A)-H(22A)	0.9500
C(3A)-H(3A)	0.9500	C(22A)-C(23A)	1.379(14)
C(3A)-C(4A)	1.378(13)	C(23A)-H(23A)	0.9500
C(4A)-H(4A)	0.9500	C(23A)-C(24A)	1.370(12)
C(4A)-C(5A)	1.396(14)	C(24A)-H(24A)	0.9500
C(5A)-C(6A)	1.388(13)	C(24A)-C(25A)	1.392(11)
C(5A)-C(8A)	1.468(17)	C(26A)-H(26A)	1.0000
C(6A)-H(6A)	0.9500	C(26A)-C(27A)	1.59(7)
C(6A)-C(7A)	1.379(14)	C(26A)-C(31A)	1.50(3)
C(7A)-H(7A)	0.9500	C(27A)-H(27A)	1.0000
C(8A)-C(10A)	1.518(18)	C(27A)-C(28A)	1.40(8)
C(10A)-H(10A)	0.9500	C(33)-C(34)	1.383(4)
C(10A)-C(11A)	1.272(15)	C(33)-C(38)	1.376(4)
C(11A)-H(11A)	0.9500	C(33)-N(30)	1.443(17)
C(11A)-C(12A)	1.521(15)	C(34)-H(34)	0.9500
C(12A)-C(13A)	1.555(14)	C(34)-C(35)	1.387(4)
C(12A)-C(25A)	1.532(13)	C(35)-H(35)	0.9500
C(12A)-C(26A)	1.597(18)	C(35)-C(36)	1.384(5)
C(13A)-C(14A)	1.390(11)	C(36)-H(36)	0.9500
C(13A)-C(18A)	1.392(13)	C(36)-C(37)	1.378(5)
C(14A)-H(14A)	0.9500	C(37)-H(37)	0.9500
C(14A)-C(15A)	1.388(12)	C(37)-C(38)	1.386(4)
C(15A)-H(15A)	0.9500	C(38)-H(38)	0.9500
C(15A)-C(16A)	1.340(14)	O(9)-C(8)	1.227(11)
C(16A)-H(16A)	0.9500	O(29)-C(28)	1.18(5)
C(16A)-C(17A)	1.397(12)	O(32)-C(31)	1.19(2)
C(17A)-H(17A)	0.9500	N(30)-C(28)	1.45(4)

N(30)-C(31)	1.36(2)	C(22)-C(21)	1.3900
C(4)-H(4)	0.9500	C(21)-H(21)	0.9500
C(4)-C(3)	1.3900	C(21)-C(20)	1.3900
C(4)-C(5)	1.3900	C(20)-C(25)	1.3900
C(3)-H(3)	0.9500	C(25)-C(24)	1.3900
C(3)-C(2)	1.3900	C(24)-H(24)	0.9500
C(2)-C(7)	1.3900	C(26)-H(26)	1.0000
C(7)-H(7)	0.9500	C(26)-C(27)	1.54(5)
C(7)-C(6)	1.3900	C(26)-C(31)	1.538(15)
C(6)-H(6)	0.9500	C(27)-H(27)	1.0000
C(6)-C(5)	1.3900	C(27)-C(28)	1.58(5)
C(5)-C(8)	1.489(9)		
C(8)-C(10)	1.462(10)	C(28A)-N(30A)-C(31A)	104(3)
C(10)-H(10)	0.9500	C(28A)-N(30A)-C(33)	127(3)
C(10)-C(11)	1.335(9)	C(33)-N(30A)-C(31A)	122(2)
C(11)-H(11)	0.9500	C(3A)-C(2A)-Br(1A)	119.6(9)
C(11)-C(12)	1.493(10)	C(7A)-C(2A)-Br(1A)	119.1(11)
C(12)-C(13)	1.531(7)	C(7A)-C(2A)-C(3A)	121.3(11)
C(12)-C(25)	1.538(6)	C(2A)-C(3A)-H(3A)	120.0
C(12)-C(26)	1.562(11)	C(2A)-C(3A)-C(4A)	119.9(9)
C(18)-C(17)	1.3900	C(4A)-C(3A)-H(3A)	120.0
C(18)-C(13)	1.3900	C(3A)-C(4A)-H(4A)	120.2
C(18)-C(19)	1.531(18)	C(3A)-C(4A)-C(5A)	119.6(9)
C(17)-H(17)	0.9500	C(5A)-C(4A)-H(4A)	120.2
C(17)-C(16)	1.3900	C(4A)-C(5A)-C(8A)	120.7(11)
C(16)-H(16)	0.9500	C(6A)-C(5A)-C(4A)	118.5(11)
C(16)-C(15)	1.3900	C(6A)-C(5A)-C(8A)	120.8(12)
C(15)-H(15)	0.9500	C(5A)-C(6A)-H(6A)	119.7
C(15)-C(14)	1.3900	C(7A)-C(6A)-C(5A)	120.6(10)
C(14)-H(14)	0.9500	C(7A)-C(6A)-H(6A)	119.7
C(14)-C(13)	1.3900	C(2A)-C(7A)-C(6A)	119.8(10)
C(19)-H(19)	1.0000	C(2A)-C(7A)-H(7A)	120.1
C(19)-C(20)	1.494(18)	C(6A)-C(7A)-H(7A)	120.1
C(19)-C(27)	1.62(4)	O(9A)-C(8A)-C(5A)	122.6(14)
C(23)-H(23)	0.9500	O(9A)-C(8A)-C(10A)	119.3(14)
C(23)-C(22)	1.3900	C(5A)-C(8A)-C(10A)	118.1(11)
C(23)-C(24)	1.3900	C(8A)-C(10A)-H(10A)	119.7
C(22)-H(22)	0.9500	C(11A)-C(10A)-C(8A)	120.6(10)

C(11A)-C(10A)-H(10A)	119.7	C(22A)-C(21A)-C(20A)	116.5(10)
C(10A)-C(11A)-H(11A)	115.5	C(22A)-C(21A)-H(21A)	121.8
C(10A)-C(11A)-C(12A)	129.1(10)	C(21A)-C(22A)-H(22A)	119.2
C(12A)-C(11A)-H(11A)	115.5	C(23A)-C(22A)-C(21A)	121.7(9)
C(11A)-C(12A)-C(13A)	109.3(8)	C(23A)-C(22A)-H(22A)	119.2
C(11A)-C(12A)-C(25A)	113.2(9)	C(22A)-C(23A)-H(23A)	118.9
C(11A)-C(12A)-C(26A)	116.9(11)	C(24A)-C(23A)-C(22A)	122.2(8)
C(13A)-C(12A)-C(26A)	103.5(11)	C(24A)-C(23A)-H(23A)	118.9
C(25A)-C(12A)-C(13A)	108.8(8)	C(23A)-C(24A)-H(24A)	121.4
C(25A)-C(12A)-C(26A)	104.4(11)	C(23A)-C(24A)-C(25A)	117.2(9)
C(14A)-C(13A)-C(12A)	125.0(9)	C(25A)-C(24A)-H(24A)	121.4
C(14A)-C(13A)-C(18A)	119.7(8)	C(20A)-C(25A)-C(12A)	113.9(9)
C(18A)-C(13A)-C(12A)	115.3(8)	C(24A)-C(25A)-C(12A)	124.9(9)
C(13A)-C(14A)-H(14A)	121.2	C(24A)-C(25A)-C(20A)	120.5(8)
C(15A)-C(14A)-C(13A)	117.5(9)	C(12A)-C(26A)-H(26A)	109.9
C(15A)-C(14A)-H(14A)	121.2	C(27A)-C(26A)-C(12A)	116(3)
C(14A)-C(15A)-H(15A)	119.0	C(27A)-C(26A)-H(26A)	109.9
C(16A)-C(15A)-C(14A)	122.1(8)	C(31A)-C(26A)-C(12A)	111.8(13)
C(16A)-C(15A)-H(15A)	119.0	C(31A)-C(26A)-H(26A)	109.9
C(15A)-C(16A)-H(16A)	119.7	C(31A)-C(26A)-C(27A)	98(2)
C(15A)-C(16A)-C(17A)	120.6(10)	C(19A)-C(27A)-C(26A)	96(3)
C(17A)-C(16A)-H(16A)	119.7	C(19A)-C(27A)-H(27A)	112.6
C(16A)-C(17A)-H(17A)	120.8	C(26A)-C(27A)-H(27A)	112.6
C(18A)-C(17A)-C(16A)	118.3(10)	C(28A)-C(27A)-C(19A)	114(5)
C(18A)-C(17A)-H(17A)	120.8	C(28A)-C(27A)-C(26A)	108(4)
C(13A)-C(18A)-C(19A)	103.6(14)	C(28A)-C(27A)-H(27A)	112.6
C(17A)-C(18A)-C(13A)	121.6(8)	O(29A)-C(28A)-N(30A)	118(4)
C(17A)-C(18A)-C(19A)	133.7(15)	O(29A)-C(28A)-C(27A)	126(5)
C(18A)-C(19A)-H(19A)	103.5	N(30A)-C(28A)-C(27A)	115(5)
C(18A)-C(19A)-C(20A)	105(2)	O(32A)-C(31A)-N(30A)	121(2)
C(20A)-C(19A)-H(19A)	103.5	O(32A)-C(31A)-C(26A)	126.3(19)
C(27A)-C(19A)-C(18A)	123(3)	C(26A)-C(31A)-N(30A)	112(2)
C(27A)-C(19A)-H(19A)	103.5	C(34)-C(33)-N(30A)	122.6(12)
C(27A)-C(19A)-C(20A)	115(3)	C(34)-C(33)-N(30)	115.0(7)
C(21A)-C(20A)-C(19A)	131.4(14)	C(38)-C(33)-N(30A)	115.6(12)
C(21A)-C(20A)-C(25A)	121.9(8)	C(38)-C(33)-C(34)	121.5(3)
C(25A)-C(20A)-C(19A)	104.9(13)	C(38)-C(33)-N(30)	123.4(7)
C(20A)-C(21A)-H(21A)	121.8	C(33)-C(34)-H(34)	120.7

C(33)-C(34)-C(35)	118.7(3)	C(8)-C(10)-H(10)	117.2
C(35)-C(34)-H(34)	120.7	C(11)-C(10)-C(8)	125.6(6)
C(34)-C(35)-H(35)	119.8	C(11)-C(10)-H(10)	117.2
C(36)-C(35)-C(34)	120.5(3)	C(10)-C(11)-H(11)	118.1
C(36)-C(35)-H(35)	119.8	C(10)-C(11)-C(12)	123.9(6)
C(35)-C(36)-H(36)	120.1	C(12)-C(11)-H(11)	118.1
C(37)-C(36)-C(35)	119.8(3)	C(11)-C(12)-C(13)	112.8(5)
C(37)-C(36)-H(36)	120.1	C(11)-C(12)-C(25)	115.1(5)
C(36)-C(37)-H(37)	119.8	C(11)-C(12)-C(26)	111.8(7)
C(36)-C(37)-C(38)	120.4(3)	C(13)-C(12)-C(25)	106.8(4)
C(38)-C(37)-H(37)	119.8	C(13)-C(12)-C(26)	105.7(7)
C(33)-C(38)-C(37)	119.1(3)	C(25)-C(12)-C(26)	103.9(6)
C(33)-C(38)-H(38)	120.5	C(17)-C(18)-C(13)	120.0
C(37)-C(38)-H(38)	120.5	C(17)-C(18)-C(19)	120.1(7)
C(33)-N(30)-C(28)	119.7(16)	C(13)-C(18)-C(19)	119.8(7)
C(31)-N(30)-C(33)	121.5(15)	C(18)-C(17)-H(17)	120.0
C(31)-N(30)-C(28)	116.3(17)	C(16)-C(17)-C(18)	120.0
C(3)-C(4)-H(4)	120.0	C(16)-C(17)-H(17)	120.0
C(3)-C(4)-C(5)	120.0	C(17)-C(16)-H(16)	120.0
C(5)-C(4)-H(4)	120.0	C(17)-C(16)-C(15)	120.0
C(4)-C(3)-H(3)	120.0	C(15)-C(16)-H(16)	120.0
C(2)-C(3)-C(4)	120.0	C(16)-C(15)-H(15)	120.0
C(2)-C(3)-H(3)	120.0	C(14)-C(15)-C(16)	120.0
C(3)-C(2)-Br(1)	119.1(4)	C(14)-C(15)-H(15)	120.0
C(3)-C(2)-C(7)	120.0	C(15)-C(14)-H(14)	120.0
C(7)-C(2)-Br(1)	120.9(4)	C(15)-C(14)-C(13)	120.0
C(2)-C(7)-H(7)	120.0	C(13)-C(14)-H(14)	120.0
C(2)-C(7)-C(6)	120.0	C(18)-C(13)-C(12)	113.6(4)
C(6)-C(7)-H(7)	120.0	C(14)-C(13)-C(12)	126.4(4)
C(7)-C(6)-H(6)	120.0	C(14)-C(13)-C(18)	120.0
C(5)-C(6)-C(7)	120.0	C(18)-C(19)-H(19)	118.0
C(5)-C(6)-H(6)	120.0	C(18)-C(19)-C(27)	94.9(14)
C(4)-C(5)-C(8)	116.4(6)	C(20)-C(19)-C(18)	107.1(13)
C(6)-C(5)-C(4)	120.0	C(20)-C(19)-H(19)	118.0
C(6)-C(5)-C(8)	123.2(5)	C(20)-C(19)-C(27)	96.8(16)
O(9)-C(8)-C(5)	119.4(9)	C(27)-C(19)-H(19)	118.0
O(9)-C(8)-C(10)	119.6(8)	C(22)-C(23)-H(23)	120.0
C(10)-C(8)-C(5)	121.0(8)	C(22)-C(23)-C(24)	120.0

C(24)-C(23)-H(23)	120.0	C(27)-C(26)-C(12)	106.8(14)
C(23)-C(22)-H(22)	120.0	C(27)-C(26)-H(26)	110.6
C(23)-C(22)-C(21)	120.0	C(31)-C(26)-C(12)	110.1(8)
C(21)-C(22)-H(22)	120.0	C(31)-C(26)-H(26)	110.6
C(22)-C(21)-H(21)	120.0	C(31)-C(26)-C(27)	108.0(14)
C(20)-C(21)-C(22)	120.0	C(19)-C(27)-H(27)	109.4
C(20)-C(21)-H(21)	120.0	C(26)-C(27)-C(19)	118.5(19)
C(21)-C(20)-C(19)	118.9(6)	C(26)-C(27)-H(27)	109.4
C(21)-C(20)-C(25)	120.0	C(26)-C(27)-C(28)	103(2)
C(25)-C(20)-C(19)	121.1(6)	C(28)-C(27)-C(19)	106(2)
C(20)-C(25)-C(12)	112.9(4)	C(28)-C(27)-H(27)	109.4
C(24)-C(25)-C(12)	126.7(4)	O(29)-C(28)-N(30)	132(4)
C(24)-C(25)-C(20)	120.0	O(29)-C(28)-C(27)	124(4)
C(23)-C(24)-H(24)	120.0	N(30)-C(28)-C(27)	104(2)
C(25)-C(24)-C(23)	120.0	O(32)-C(31)-N(30)	126.4(15)
C(25)-C(24)-H(24)	120.0	O(32)-C(31)-C(26)	126.9(12)
C(12)-C(26)-H(26)	110.6	N(30)-C(31)-C(26)	105.8(12)

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd814b. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	20(1)	26(1)	27(1)	4(1)	5(1)	0(1)
Br(1A)	22(1)	38(1)	28(1)	6(1)	5(1)	4(1)
O(9A)	30(5)	16(7)	43(5)	-9(6)	14(4)	-4(6)
O(29A)	17(6)	17(6)	26(5)	0(4)	1(5)	6(4)
O(32A)	17(4)	13(8)	51(7)	-3(6)	13(4)	-7(6)
N(30A)	20(2)	21(4)	26(4)	1(3)	2(2)	1(3)
C(2A)	24(4)	21(3)	16(3)	5(3)	5(3)	3(3)
C(3A)	14(3)	29(3)	22(4)	-1(3)	11(3)	0(3)
C(4A)	25(4)	24(3)	16(4)	-3(3)	11(3)	-6(3)
C(5A)	16(4)	25(4)	22(3)	2(3)	11(3)	4(3)
C(6A)	12(4)	18(3)	19(3)	-3(3)	4(3)	-1(3)
C(7A)	18(4)	25(3)	21(3)	1(3)	9(3)	5(3)
C(8A)	30(6)	26(7)	22(6)	-4(4)	6(4)	-7(5)
C(10A)	24(4)	21(4)	27(4)	2(3)	0(3)	-2(3)
C(11A)	20(4)	23(4)	23(4)	6(3)	1(3)	-2(3)
C(12A)	21(3)	14(3)	19(3)	0(3)	7(3)	-2(3)
C(13A)	13(3)	21(3)	17(3)	3(3)	3(2)	-3(2)
C(14A)	19(3)	18(3)	17(3)	-1(3)	6(3)	-1(3)
C(15A)	15(3)	25(3)	16(3)	-2(3)	1(3)	-1(3)
C(16A)	17(3)	21(3)	20(3)	1(3)	3(3)	0(3)
C(17A)	14(3)	18(3)	15(3)	1(3)	6(2)	0(2)
C(18A)	18(3)	14(3)	15(3)	0(2)	6(3)	2(2)
C(19A)	19(2)	16(2)	18(4)	-1(3)	2(2)	-1(2)
C(20A)	15(3)	15(3)	17(3)	-2(3)	8(2)	-3(2)
C(21A)	17(3)	15(3)	19(3)	-4(3)	8(3)	0(2)
C(22A)	22(3)	20(3)	19(3)	-4(3)	5(3)	0(3)
C(23A)	17(3)	20(3)	20(3)	-2(3)	5(3)	1(3)
C(24A)	21(3)	17(3)	22(3)	-2(3)	8(3)	-1(3)
C(25A)	21(3)	16(3)	16(3)	-1(3)	3(2)	-6(2)
C(26A)	14(4)	25(5)	24(4)	0(4)	3(3)	-5(4)
C(27A)	19(3)	23(4)	28(1)	-3(3)	2(2)	0(3)
C(28A)	21(2)	24(3)	26(4)	0(3)	5(3)	-1(2)
C(31A)	19(3)	24(4)	28(3)	0(3)	7(2)	-9(3)

C(33)	19(1)	26(2)	39(2)	-9(1)	2(1)	-1(1)
C(34)	26(2)	29(2)	37(2)	-9(1)	6(1)	0(1)
C(35)	34(2)	35(2)	39(2)	-11(2)	-2(2)	-4(2)
C(36)	24(2)	32(2)	62(2)	-12(2)	-2(2)	-5(1)
C(37)	24(2)	36(2)	61(2)	2(2)	9(2)	-2(1)
C(38)	29(2)	38(2)	42(2)	-1(2)	7(2)	0(2)
O(9)	31(2)	18(3)	43(3)	-6(3)	14(2)	-8(3)
O(29)	18(10)	12(8)	38(12)	4(7)	1(8)	3(7)
O(32)	24(3)	19(6)	39(4)	-7(4)	14(2)	-10(4)
N(30)	20(2)	21(4)	26(4)	1(3)	2(2)	1(3)
C(4)	14(3)	19(2)	25(3)	1(2)	7(2)	-1(2)
C(3)	17(4)	23(3)	20(2)	-2(2)	5(3)	0(3)
C(2)	17(3)	24(3)	19(3)	8(2)	8(2)	3(2)
C(7)	23(4)	29(2)	19(3)	-9(2)	10(3)	-7(3)
C(6)	19(3)	29(3)	22(3)	-2(2)	10(2)	-2(2)
C(5)	15(3)	20(3)	23(3)	0(2)	7(2)	-1(2)
C(8)	16(4)	28(6)	21(4)	2(3)	6(3)	-2(4)
C(10)	21(2)	25(3)	21(2)	-1(2)	7(2)	1(2)
C(11)	22(3)	21(3)	20(3)	-2(2)	4(2)	-7(2)
C(12)	16(2)	19(3)	16(2)	-1(2)	4(2)	-4(2)
C(18)	14(2)	18(2)	19(3)	2(2)	2(2)	0(2)
C(17)	18(3)	19(2)	21(3)	0(2)	7(2)	5(2)
C(16)	14(2)	22(2)	22(3)	3(2)	2(2)	2(2)
C(15)	19(3)	21(2)	20(3)	-4(2)	0(2)	1(2)
C(14)	19(2)	22(2)	18(2)	1(2)	6(2)	-1(2)
C(13)	17(2)	20(2)	16(2)	1(2)	6(2)	-2(2)
C(19)	19(2)	16(2)	18(4)	-1(3)	2(2)	-1(2)
C(23)	19(2)	23(2)	27(3)	0(2)	5(2)	4(2)
C(22)	23(3)	25(3)	19(3)	-1(2)	2(2)	1(2)
C(21)	22(3)	18(3)	22(3)	-7(2)	10(2)	-1(2)
C(20)	17(2)	17(2)	19(3)	-1(2)	5(2)	-5(2)
C(25)	18(2)	17(2)	19(2)	-1(2)	6(2)	-4(2)
C(24)	22(2)	22(2)	21(3)	-2(2)	5(2)	-2(2)
C(26)	22(3)	21(4)	26(3)	-3(3)	5(2)	-6(3)
C(27)	19(3)	23(4)	28(1)	-3(3)	2(2)	0(3)
C(28)	21(2)	24(3)	26(4)	0(3)	5(3)	-1(2)
C(31)	19(3)	24(4)	28(3)	0(3)	7(2)	-9(3)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd814b.

	x	y	z	U(eq)
H(3A)	10036	3822	5364	25
H(4A)	8562	3875	5692	25
H(6A)	7404	147	4539	20
H(7A)	8823	312	4166	25
H(10A)	6703	4601	5446	31
H(11A)	6104	2455	6192	28
H(14A)	4277	2867	5575	21
H(15A)	2496	3006	5389	23
H(16A)	1760	4659	5945	24
H(17A)	2754	6591	6619	18
H(19A)	4595	7937	6967	22
H(21A)	5773	7308	8086	20
H(22A)	7269	5923	8615	24
H(23A)	8046	4008	8151	23
H(24A)	7430	3449	7133	23
H(26A)	5657	6831	5659	25
H(27A)	4901	8883	6085	29
H(34)	7875	9065	7840	38
H(35)	9407	10295	8390	46
H(36)	10528	11440	7900	51
H(37)	10135	11334	6861	49
H(38)	8597	10144	6304	44
H(4)	7170	190	4542	23
H(3)	8598	506	4174	24
H(7)	9814	3791	5498	27
H(6)	8386	3476	5865	27
H(10)	6260	2031	6216	27
H(11)	6679	4657	5548	26
H(17)	2860	6461	6511	23
H(16)	1912	4526	5831	24
H(15)	2730	2830	5288	25
H(14)	4496	3069	5426	23

H(19)	4513	7734	7096	22
H(23)	8199	3942	7999	28
H(22)	7357	5678	8516	28
H(21)	5869	7075	7997	24
H(24)	7551	3603	6963	26
H(26)	5694	7319	5598	28
H(27)	4824	8750	6112	29

Table 7. Torsion angles [°] for tcd814b.

Br(1)-C(2)-C(7)-C(6)	178.1(7)	C(13A)-C(12A)-C(25A)-C(20A)	-50.2(12)
Br(1A)-C(2A)-C(3A)-C(4A)	174.4(11)	C(13A)-C(12A)-C(25A)-C(24A)	139.3(11)
Br(1A)-C(2A)-C(7A)-C(6A)	-177.5(12)	C(13A)-C(12A)-C(26A)-C(27A)	57(3)
O(9A)-C(8A)-C(10A)-C(11A)	33.3(16)	C(13A)-C(12A)-C(26A)-C(31A)	168.9(13)
N(30A)-C(33)-C(34)-C(35)	-172.6(12)	C(13A)-C(14A)-C(15A)-C(16A)	-4.9(13)
N(30A)-C(33)-C(38)-C(37)	173.5(11)	C(13A)-C(18A)-C(19A)-C(20A)	-72.0(15)
C(2A)-C(3A)-C(4A)-C(5A)	4.8(14)	C(13A)-C(18A)-C(19A)-C(27A)	63(4)
C(3A)-C(2A)-C(7A)-C(6A)	2.7(13)	C(14A)-C(13A)-C(18A)-C(17A)	2.2(14)
C(3A)-C(4A)-C(5A)-C(6A)	-0.8(13)	C(14A)-C(13A)-C(18A)-C(19A)	-167.5(13)
C(3A)-C(4A)-C(5A)-C(8A)	179.8(13)	C(14A)-C(15A)-C(16A)-C(17A)	5.1(14)
C(4A)-C(5A)-C(6A)-C(7A)	-2.4(13)	C(15A)-C(16A)-C(17A)-C(18A)	-1.4(13)
C(4A)-C(5A)-C(8A)-O(9A)	-151.7(13)	C(16A)-C(17A)-C(18A)-C(13A)	-2.2(13)
C(4A)-C(5A)-C(8A)-C(10A)	27.8(17)	C(16A)-C(17A)-C(18A)-C(19A)	163.9(17)
C(5A)-C(6A)-C(7A)-C(2A)	1.4(13)	C(17A)-C(18A)-C(19A)-C(20A)	120.2(18)
C(5A)-C(8A)-C(10A)-C(11A)	-146.3(11)	C(17A)-C(18A)-C(19A)-C(27A)	-105(3)
C(6A)-C(5A)-C(8A)-O(9A)	28.9(19)	C(18A)-C(13A)-C(14A)-C(15A)	1.3(13)
C(6A)-C(5A)-C(8A)-C(10A)	-151.6(11)	C(18A)-C(19A)-C(20A)-C(21A)	-125.3(17)
C(7A)-C(2A)-C(3A)-C(4A)	-5.9(14)	C(18A)-C(19A)-C(20A)-C(25A)	70.3(16)
C(8A)-C(5A)-C(6A)-C(7A)	177.0(13)	C(18A)-C(19A)-C(27A)-C(26A)	-64(4)
C(8A)-C(10A)-C(11A)-C(12A)	-176.6(10)	C(18A)-C(19A)-C(27A)-C(28A)	-177(4)
C(10A)-C(11A)-C(12A)-C(13A)	112.8(10)	C(19A)-C(20A)-C(21A)-C(22A)	-161.6(18)
C(10A)-C(11A)-C(12A)-C(25A)	-125.7(10)	C(19A)-C(20A)-C(25A)-C(12A)	-6.1(16)
C(10A)-C(11A)-C(12A)-C(26A)	-4.3(16)	C(19A)-C(20A)-C(25A)-C(24A)	164.8(15)
C(11A)-C(12A)-C(13A)-C(14A)	-8.3(13)	C(19A)-C(27A)-C(28A)-O(29A)	-60(7)
C(11A)-C(12A)-C(13A)-C(18A)	172.6(9)	C(19A)-C(27A)-C(28A)-N(30A)	118(4)
C(11A)-C(12A)-C(25A)-C(20A)	-172.0(9)	C(20A)-C(19A)-C(27A)-C(26A)	67(4)
C(11A)-C(12A)-C(25A)-C(24A)	17.6(14)	C(20A)-C(19A)-C(27A)-C(28A)	-46(6)
C(11A)-C(12A)-C(26A)-C(27A)	177(3)	C(20A)-C(21A)-C(22A)-C(23A)	-0.5(13)
C(11A)-C(12A)-C(26A)-C(31A)	-70.9(17)	C(21A)-C(20A)-C(25A)-C(12A)	-172.4(10)
C(12A)-C(13A)-C(14A)-C(15A)	-177.8(9)	C(21A)-C(20A)-C(25A)-C(24A)	-1.5(14)
C(12A)-C(13A)-C(18A)-C(17A)	-178.6(10)	C(21A)-C(22A)-C(23A)-C(24A)	1.3(14)
C(12A)-C(13A)-C(18A)-C(19A)	11.7(15)	C(22A)-C(23A)-C(24A)-C(25A)	-2.1(14)
C(12A)-C(26A)-C(27A)-C(19A)	-3(4)	C(23A)-C(24A)-C(25A)-C(12A)	172.0(10)
C(12A)-C(26A)-C(27A)-C(28A)	115(4)	C(23A)-C(24A)-C(25A)-C(20A)	2.2(14)
C(12A)-C(26A)-C(31A)-O(32A)	64(2)	C(25A)-C(12A)-C(13A)-C(14A)	-132.4(10)
C(12A)-C(26A)-C(31A)-N(30A)	-126.7(18)	C(25A)-C(12A)-C(13A)-C(18A)	48.5(12)

C(25A)-C(12A)-C(26A)-C(27A)	-57(3)	C(38)-C(33)-C(34)-C(35)	0.8(5)
C(25A)-C(12A)-C(26A)-C(31A)	55.0(17)	C(38)-C(33)-N(30)-C(28)	-121.9(16)
C(25A)-C(20A)-C(21A)-C(22A)	0.6(13)	C(38)-C(33)-N(30)-C(31)	39.6(13)
C(26A)-C(12A)-C(13A)-C(14A)	117.0(13)	O(9)-C(8)-C(10)-C(11)	-143.2(7)
C(26A)-C(12A)-C(13A)-C(18A)	-62.1(13)	N(30)-C(33)-C(34)-C(35)	177.9(7)
C(26A)-C(12A)-C(25A)-C(20A)	59.8(14)	N(30)-C(33)-C(38)-C(37)	-177.2(7)
C(26A)-C(12A)-C(25A)-C(24A)	-110.7(14)	C(4)-C(3)-C(2)-Br(1)	-178.1(6)
C(26A)-C(27A)-C(28A)-O(29A)	-165(4)	C(4)-C(3)-C(2)-C(7)	0.0
C(26A)-C(27A)-C(28A)-N(30A)	13(6)	C(4)-C(5)-C(8)-O(9)	21.5(10)
C(27A)-C(19A)-C(20A)-C(21A)	95(3)	C(4)-C(5)-C(8)-C(10)	-161.3(6)
C(27A)-C(19A)-C(20A)-C(25A)	-69(3)	C(3)-C(4)-C(5)-C(6)	0.0
C(27A)-C(26A)-C(31A)-O(32A)	-174(3)	C(3)-C(4)-C(5)-C(8)	-172.4(7)
C(27A)-C(26A)-C(31A)-N(30A)	-4(3)	C(3)-C(2)-C(7)-C(6)	0.0
C(28A)-N(30A)-C(31A)-O(32A)	-178(3)	C(2)-C(7)-C(6)-C(5)	0.0
C(28A)-N(30A)-C(31A)-C(26A)	12(3)	C(7)-C(6)-C(5)-C(4)	0.0
C(28A)-N(30A)-C(33)-C(34)	38(4)	C(7)-C(6)-C(5)-C(8)	171.9(8)
C(28A)-N(30A)-C(33)-C(38)	-136(3)	C(6)-C(5)-C(8)-O(9)	-150.6(7)
C(31A)-N(30A)-C(28A)-O(29A)	164(4)	C(6)-C(5)-C(8)-C(10)	26.6(11)
C(31A)-N(30A)-C(28A)-C(27A)	-15(5)	C(5)-C(4)-C(3)-C(2)	0.0
C(31A)-N(30A)-C(33)-C(34)	-107(2)	C(5)-C(8)-C(10)-C(11)	39.6(10)
C(31A)-N(30A)-C(33)-C(38)	79(2)	C(8)-C(10)-C(11)-C(12)	169.1(6)
C(31A)-C(26A)-C(27A)-C(19A)	-122(2)	C(10)-C(11)-C(12)-C(13)	-63.9(7)
C(31A)-C(26A)-C(27A)-C(28A)	-5(4)	C(10)-C(11)-C(12)-C(25)	59.0(7)
C(33)-N(30A)-C(28A)-O(29A)	14(6)	C(10)-C(11)-C(12)-C(26)	177.1(7)
C(33)-N(30A)-C(28A)-C(27A)	-165(3)	C(11)-C(12)-C(13)-C(18)	-179.8(4)
C(33)-N(30A)-C(31A)-O(32A)	-27(3)	C(11)-C(12)-C(13)-C(14)	-1.0(7)
C(33)-N(30A)-C(31A)-C(26A)	163.1(18)	C(11)-C(12)-C(25)-C(20)	-179.8(5)
C(33)-C(34)-C(35)-C(36)	-0.4(5)	C(11)-C(12)-C(25)-C(24)	8.2(8)
C(33)-N(30)-C(28)-O(29)	6(5)	C(11)-C(12)-C(26)-C(27)	176.5(15)
C(33)-N(30)-C(28)-C(27)	179.3(16)	C(11)-C(12)-C(26)-C(31)	-66.5(9)
C(33)-N(30)-C(31)-O(32)	13.7(18)	C(12)-C(25)-C(24)-C(23)	171.4(6)
C(33)-N(30)-C(31)-C(26)	-176.7(8)	C(12)-C(26)-C(27)-C(19)	5(3)
C(34)-C(33)-C(38)-C(37)	-0.3(5)	C(12)-C(26)-C(27)-C(28)	122.1(18)
C(34)-C(33)-N(30)-C(28)	61.1(16)	C(12)-C(26)-C(31)-O(32)	59.1(14)
C(34)-C(33)-N(30)-C(31)	-137.5(10)	C(12)-C(26)-C(31)-N(30)	-110.4(10)
C(34)-C(35)-C(36)-C(37)	-0.4(5)	C(18)-C(17)-C(16)-C(15)	0.0
C(35)-C(36)-C(37)-C(38)	0.9(5)	C(18)-C(19)-C(20)-C(21)	-141.4(6)
C(36)-C(37)-C(38)-C(33)	-0.6(5)	C(18)-C(19)-C(20)-C(25)	38.9(12)

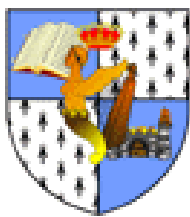
C(18)-C(19)-C(27)-C(26)	-58(3)	C(22)-C(21)-C(20)-C(25)	0.0
C(18)-C(19)-C(27)-C(28)	-173(2)	C(21)-C(20)-C(25)-C(12)	-172.6(5)
C(17)-C(18)-C(13)-C(12)	178.9(5)	C(21)-C(20)-C(25)-C(24)	0.0
C(17)-C(18)-C(13)-C(14)	0.0	C(20)-C(19)-C(27)-C(26)	50(3)
C(17)-C(18)-C(19)-C(20)	135.6(7)	C(20)-C(19)-C(27)-C(28)	-65(2)
C(17)-C(18)-C(19)-C(27)	-125.8(14)	C(20)-C(25)-C(24)-C(23)	0.0
C(17)-C(16)-C(15)-C(14)	0.0	C(25)-C(12)-C(13)-C(18)	52.8(5)
C(16)-C(15)-C(14)-C(13)	0.0	C(25)-C(12)-C(13)-C(14)	-128.4(5)
C(15)-C(14)-C(13)-C(12)	-178.7(5)	C(25)-C(12)-C(26)-C(27)	-58.8(17)
C(15)-C(14)-C(13)-C(18)	0.0	C(25)-C(12)-C(26)-C(31)	58.2(9)
C(13)-C(12)-C(25)-C(20)	-53.7(6)	C(24)-C(23)-C(22)-C(21)	0.0
C(13)-C(12)-C(25)-C(24)	134.3(5)	C(26)-C(12)-C(13)-C(18)	-57.3(7)
C(13)-C(12)-C(26)-C(27)	53.4(17)	C(26)-C(12)-C(13)-C(14)	121.5(7)
C(13)-C(12)-C(26)-C(31)	170.4(7)	C(26)-C(12)-C(25)-C(20)	57.7(7)
C(13)-C(18)-C(17)-C(16)	0.0	C(26)-C(12)-C(25)-C(24)	-114.3(8)
C(13)-C(18)-C(19)-C(20)	-39.7(11)	C(26)-C(27)-C(28)-O(29)	163(3)
C(13)-C(18)-C(19)-C(27)	58.9(16)	C(26)-C(27)-C(28)-N(30)	-11(3)
C(19)-C(18)-C(17)-C(16)	-175.3(9)	C(27)-C(19)-C(20)-C(21)	121.4(13)
C(19)-C(18)-C(13)-C(12)	-5.8(9)	C(27)-C(19)-C(20)-C(25)	-58.3(17)
C(19)-C(18)-C(13)-C(14)	175.3(9)	C(27)-C(26)-C(31)-O(32)	175.3(18)
C(19)-C(20)-C(25)-C(12)	7.1(10)	C(27)-C(26)-C(31)-N(30)	5.8(17)
C(19)-C(20)-C(25)-C(24)	179.7(10)	C(28)-N(30)-C(31)-O(32)	175.8(17)
C(19)-C(27)-C(28)-O(29)	-72(4)	C(28)-N(30)-C(31)-C(26)	-14.6(19)
C(19)-C(27)-C(28)-N(30)	114(2)	C(31)-N(30)-C(28)-O(29)	-156(4)
C(23)-C(22)-C(21)-C(20)	0.0	C(31)-N(30)-C(28)-C(27)	17(3)
C(22)-C(23)-C(24)-C(25)	0.0	C(31)-C(26)-C(27)-C(19)	-114(2)
C(22)-C(21)-C(20)-C(19)	-179.7(10)	C(31)-C(26)-C(27)-C(28)	4(2)

Table 8. Hydrogen bonds for tcd814b [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(10A)-H(10A)...O(32A)	0.95	2.51	3.04(2)	116
C(38)-H(38)...Br(1A)#1	0.95	2.91	3.545(6)	125
C(10)-H(10)...O(29)#2	0.95	2.38	3.32(5)	170

Symmetry transformations used to generate equivalent atoms:

#1 $-x+2, -y+1, -z+1$ #2 $x, y-1, z$



Small Molecule X-ray Facility School Of Chemistry



Structure Report

Filename: TCD813

Submitted by: James Mc Keown

Reference: CC56

Group: Meegan

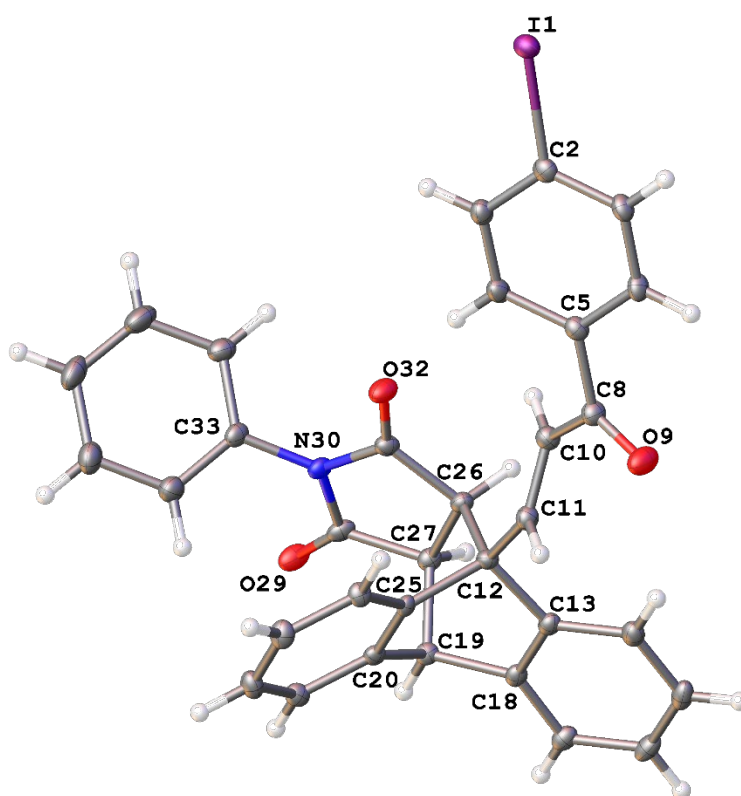


Fig. 1. Asymmetric unit of TCD813 with partial atom labelling for clarity. Atomic displacement shown at 50% probability.

6/6/17

Author: Brendan Twamley

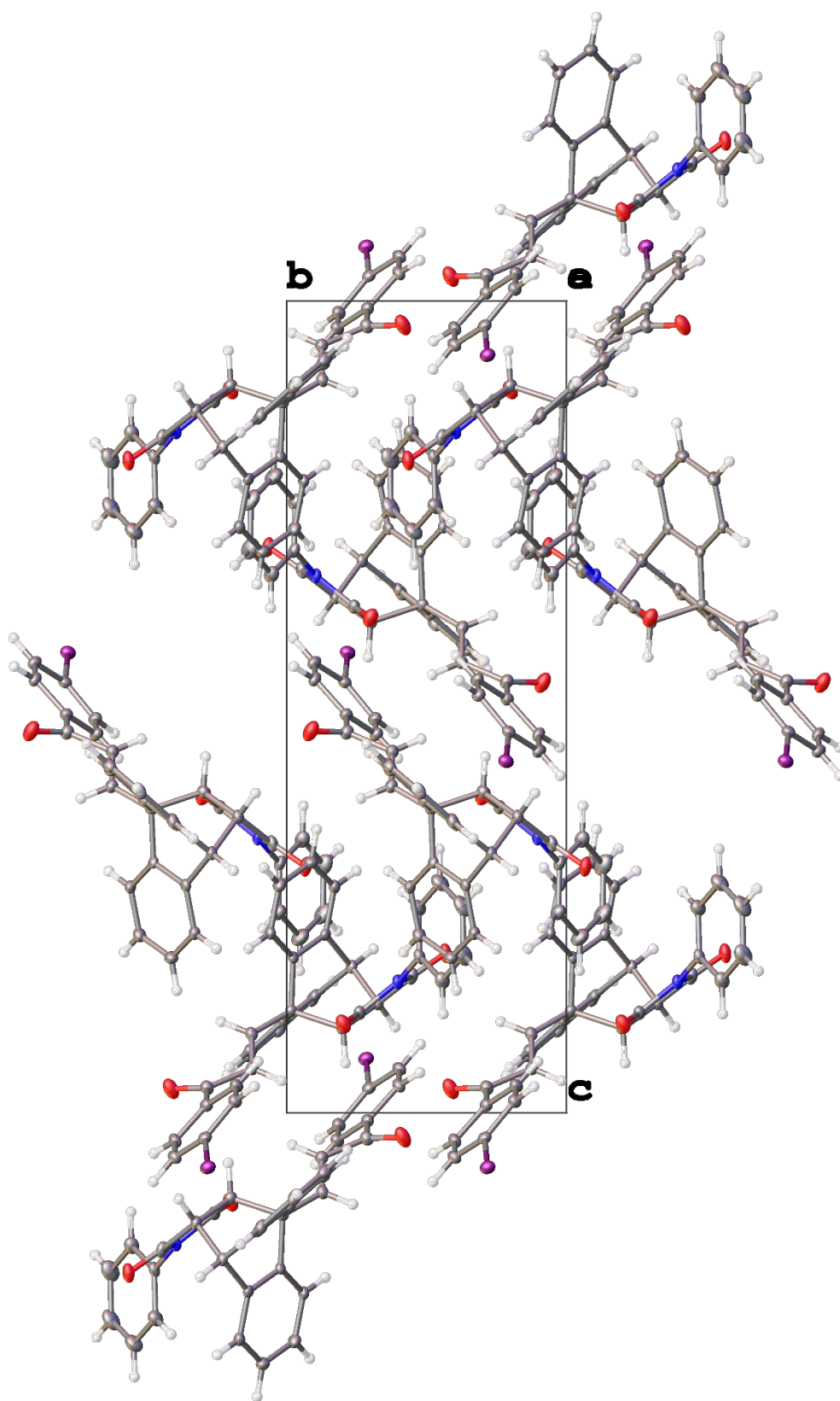


Fig. 2. Packing diagram of TCD813 showing the sinusoidal packing nature of the molecules as viewed normal to the a-axis. Atomic displacement shown at 50% probability.

Crystal Structure Report for TCD813

A clear colourless block-like specimen of $\text{C}_{33}\text{H}_{22}\text{INO}_3$, approximate dimensions 0.200 mm x 0.210 mm x 0.230 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K with an Oxford Cryosystems low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 1386 frames were collected. The total exposure time was 2.36 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 58595 reflections to a maximum θ angle of 31.09° (0.69 Å resolution), of which 8049 were independent (average redundancy 7.280, completeness = 99.9%, $R_{\text{int}} = 4.61\%$, $R_{\text{sig}} = 3.04\%$) and 6659 (82.73%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 13.7687(3)$ Å, $b = 7.9215(2)$ Å, $c = 23.9185(6)$ Å, $\beta = 105.7027(12)^\circ$, volume = $2511.40(11)$ Å³, are based upon the refinement of the XYZ-centroids of 9964 reflections above $20\sigma(I)$ with $5.365^\circ < 2\theta < 62.09^\circ$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.933. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.6965 and 0.7462.

The structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimisation with Olex2, using the space group $P2_1/n$, with $Z = 4$ for the formula unit, $\text{C}_{33}\text{H}_{22}\text{INO}_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 343 variables converged at $R1 = 3.56\%$, for the observed data and $wR2 = 11.97\%$ for all data. The goodness-of-fit was 0.913. The largest peak in the final difference electron density synthesis was $1.594 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-1.355 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.099 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.606 g/cm^3 and $F(000)$, 1216 e^- .

References: see CIF.

Table 1: Data collection details for TCD813.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Phi	50.541	8.00	0.00	0.00	54.76	1.00	180	5.00	0.71073	50	20.0	100
Omega	50.541	37.83	217.36	96.00	54.76	0.90	201	6.30	0.71073	50	20.0	100
Omega	50.541	-10.72	168.82	204.00	54.76	0.90	201	6.30	0.71073	50	20.0	100
Omega	50.541	37.83	217.36	0.00	54.76	0.90	201	6.30	0.71073	50	20.0	100
Omega	50.541	37.83	217.36	64.00	54.76	0.90	201	6.30	0.71073	50	20.0	100
Omega	50.541	37.83	217.36	96.00	54.76	0.90	201	6.30	0.71073	50	20.0	100
Omega	50.541	-10.72	168.82	0.00	54.76	0.90	201	6.30	0.71073	50	20.0	100

Crystal Data for C₃₃H₂₂INO₃ (M = 607.41 g/mol): monoclinic, space group P2₁/n (no. 14), a = 13.7687(3) Å, b = 7.9215(2) Å, c = 23.9185(6) Å, β = 105.7027(12)°, V = 2511.40(11) Å³, Z = 4, T = 100(2) K, $\mu(\text{MoK}\alpha)$ = 1.312 mm⁻¹, D_{calc} = 1.606 g/cm³, 58595 reflections measured ($5.366^\circ \leq 2\theta \leq 62.176^\circ$), 8049 unique (R_{int} = 0.0461, R_{sigma} = 0.0304) which were used in all calculations. The final R_1 was 0.0356 ($I > 2\sigma(I)$) and wR_2 was 0.1197 (all data).

Table 2. Crystal data and structure refinement for ted813.

Identification code	ted813	
Empirical formula	C ₃₃ H ₂₂ INO ₃	
Formula weight	607.41	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /n	
Unit cell dimensions	a = 13.7687(3) Å	α = 90°.
	b = 7.9215(2) Å	β = 105.7027(12)°.
	c = 23.9185(6) Å	γ = 90°.
Volume	2511.40(11) Å ³	
Z	4	
Density (calculated)	1.606 Mg/m ³	
Absorption coefficient	1.312 mm ⁻¹	
F(000)	1216	
Crystal size	0.23 x 0.21 x 0.2 mm ³	
Theta range for data collection	2.683 to 31.088°.	
Index ranges	-19 ≤ h ≤ 20, -11 ≤ k ≤ 11, -34 ≤ l ≤ 29	
Reflections collected	58595	
Independent reflections	8049 [R(int) = 0.0461]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7462 and 0.6965	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	8049 / 0 / 343	
Goodness-of-fit on F ²	0.913	
Final R indices [I > 2σ(I)]	R1 = 0.0356, wR2 = 0.1092	
R indices (all data)	R1 = 0.0498, wR2 = 0.1197	
Largest diff. peak and hole	1.594 and -1.355 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd813. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	-1205(1)	7828(1)	4331(1)	18(1)
C(2)	359(2)	7931(3)	4694(1)	16(1)
C(3)	818(2)	6855(3)	5147(1)	17(1)
C(4)	1866(2)	6802(3)	5337(1)	18(1)
C(5)	2445(2)	7878(3)	5096(1)	16(1)
C(6)	1975(2)	8997(3)	4661(1)	16(1)
C(7)	928(2)	9017(3)	4456(1)	17(1)
C(8)	3563(2)	7857(3)	5311(1)	17(1)
O(9)	4050(1)	9145(3)	5298(1)	27(1)
C(10)	4052(2)	6240(3)	5549(1)	16(1)
C(11)	4900(2)	6267(3)	5975(1)	16(1)
C(12)	5570(2)	4849(3)	6257(1)	13(1)
C(13)	6610(2)	5096(3)	6147(1)	14(1)
C(14)	6845(2)	6291(3)	5776(1)	17(1)
C(15)	7825(2)	6367(3)	5716(1)	20(1)
C(16)	8564(2)	5281(3)	6035(1)	19(1)
C(17)	8334(2)	4082(3)	6404(1)	16(1)
C(18)	7353(2)	3979(3)	6449(1)	13(1)
C(19)	6964(2)	2691(3)	6801(1)	13(1)
C(20)	6459(2)	3624(3)	7198(1)	13(1)
C(21)	6619(2)	3301(3)	7786(1)	16(1)
C(22)	6037(2)	4137(3)	8087(1)	19(1)
C(23)	5289(2)	5269(3)	7808(1)	18(1)
C(24)	5125(2)	5597(3)	7218(1)	14(1)
C(25)	5722(2)	4773(3)	6914(1)	12(1)
C(26)	5244(2)	2993(3)	6046(1)	14(1)
C(27)	6093(2)	1745(3)	6352(1)	15(1)
C(28)	5607(2)	528(3)	6681(1)	16(1)
O(29)	6001(1)	-669(2)	6968(1)	21(1)
N(30)	4608(1)	1036(3)	6614(1)	14(1)
C(31)	4321(2)	2399(3)	6235(1)	15(1)
O(32)	3483(1)	2987(2)	6100(1)	22(1)
C(33)	3977(2)	255(3)	6927(1)	16(1)

C(34)	4279(2)	303(3)	7528(1)	21(1)
C(35)	3682(2)	-473(4)	7836(1)	26(1)
C(36)	2789(2)	-1282(4)	7548(1)	29(1)
C(37)	2497(2)	-1312(4)	6945(1)	29(1)
C(38)	3089(2)	-552(3)	6631(1)	21(1)

Table 4. Bond lengths [Å] and angles [°] for *tcd813*.

I(1)-C(2)	2.094(3)	C(20)-C(25)	1.395(3)
C(2)-C(3)	1.390(3)	C(21)-H(21)	0.9500
C(2)-C(7)	1.385(3)	C(21)-C(22)	1.382(3)
C(3)-H(3)	0.9500	C(22)-H(22)	0.9500
C(3)-C(4)	1.390(4)	C(22)-C(23)	1.392(4)
C(4)-H(4)	0.9500	C(23)-H(23)	0.9500
C(4)-C(5)	1.395(4)	C(23)-C(24)	1.393(3)
C(5)-C(6)	1.385(3)	C(24)-H(24)	0.9500
C(5)-C(8)	1.485(4)	C(24)-C(25)	1.397(3)
C(6)-H(6)	0.9500	C(26)-H(26)	1.0000
C(6)-C(7)	1.391(3)	C(26)-C(27)	1.554(3)
C(7)-H(7)	0.9500	C(26)-C(31)	1.532(3)
C(8)-O(9)	1.227(3)	C(27)-H(27)	1.0000
C(8)-C(10)	1.486(3)	C(27)-C(28)	1.509(3)
C(10)-H(10)	0.9500	C(28)-O(29)	1.210(3)
C(10)-C(11)	1.326(3)	C(28)-N(30)	1.401(3)
C(11)-H(11)	0.9500	N(30)-C(31)	1.397(3)
C(11)-C(12)	1.493(3)	N(30)-C(33)	1.430(3)
C(12)-C(13)	1.537(3)	C(31)-O(32)	1.205(3)
C(12)-C(25)	1.530(3)	C(33)-C(34)	1.384(3)
C(12)-C(26)	1.580(3)	C(33)-C(38)	1.391(3)
C(13)-C(14)	1.394(3)	C(34)-H(34)	0.9500
C(13)-C(18)	1.397(3)	C(34)-C(35)	1.387(4)
C(14)-H(14)	0.9500	C(35)-H(35)	0.9500
C(14)-C(15)	1.396(4)	C(35)-C(36)	1.394(4)
C(15)-H(15)	0.9500	C(36)-H(36)	0.9500
C(15)-C(16)	1.392(4)	C(36)-C(37)	1.387(4)
C(16)-H(16)	0.9500	C(37)-H(37)	0.9500
C(16)-C(17)	1.390(3)	C(37)-C(38)	1.387(4)
C(17)-H(17)	0.9500	C(38)-H(38)	0.9500
C(17)-C(18)	1.387(3)		
C(18)-C(19)	1.510(3)	C(3)-C(2)-I(1)	119.93(19)
C(19)-H(19)	1.0000	C(7)-C(2)-I(1)	119.19(18)
C(19)-C(20)	1.513(3)	C(7)-C(2)-C(3)	120.8(2)
C(19)-C(27)	1.567(3)	C(2)-C(3)-H(3)	120.4
C(20)-C(21)	1.387(3)	C(2)-C(3)-C(4)	119.1(2)

C(4)-C(3)-H(3)	120.4	C(17)-C(16)-C(15)	120.8(2)
C(3)-C(4)-H(4)	119.8	C(17)-C(16)-H(16)	119.6
C(3)-C(4)-C(5)	120.3(2)	C(16)-C(17)-H(17)	120.5
C(5)-C(4)-H(4)	119.8	C(18)-C(17)-C(16)	118.9(2)
C(4)-C(5)-C(8)	120.3(2)	C(18)-C(17)-H(17)	120.5
C(6)-C(5)-C(4)	119.8(2)	C(13)-C(18)-C(19)	113.3(2)
C(6)-C(5)-C(8)	119.9(2)	C(17)-C(18)-C(13)	121.0(2)
C(5)-C(6)-H(6)	120.0	C(17)-C(18)-C(19)	125.7(2)
C(5)-C(6)-C(7)	120.1(2)	C(18)-C(19)-H(19)	112.4
C(7)-C(6)-H(6)	120.0	C(18)-C(19)-C(20)	108.25(18)
C(2)-C(7)-C(6)	119.7(2)	C(18)-C(19)-C(27)	105.24(19)
C(2)-C(7)-H(7)	120.1	C(20)-C(19)-H(19)	112.4
C(6)-C(7)-H(7)	120.1	C(20)-C(19)-C(27)	105.50(19)
C(5)-C(8)-C(10)	117.5(2)	C(27)-C(19)-H(19)	112.4
O(9)-C(8)-C(5)	120.3(2)	C(21)-C(20)-C(19)	125.0(2)
O(9)-C(8)-C(10)	122.2(2)	C(21)-C(20)-C(25)	120.6(2)
C(8)-C(10)-H(10)	120.2	C(25)-C(20)-C(19)	114.2(2)
C(11)-C(10)-C(8)	119.6(2)	C(20)-C(21)-H(21)	120.5
C(11)-C(10)-H(10)	120.2	C(22)-C(21)-C(20)	119.0(2)
C(10)-C(11)-H(11)	115.0	C(22)-C(21)-H(21)	120.5
C(10)-C(11)-C(12)	130.0(2)	C(21)-C(22)-H(22)	119.5
C(12)-C(11)-H(11)	115.0	C(21)-C(22)-C(23)	121.1(2)
C(11)-C(12)-C(13)	108.49(19)	C(23)-C(22)-H(22)	119.5
C(11)-C(12)-C(25)	112.95(19)	C(22)-C(23)-H(23)	119.9
C(11)-C(12)-C(26)	117.93(19)	C(22)-C(23)-C(24)	120.2(2)
C(13)-C(12)-C(26)	105.13(18)	C(24)-C(23)-H(23)	119.9
C(25)-C(12)-C(13)	107.82(17)	C(23)-C(24)-H(24)	120.6
C(25)-C(12)-C(26)	103.88(18)	C(23)-C(24)-C(25)	118.8(2)
C(14)-C(13)-C(12)	125.7(2)	C(25)-C(24)-H(24)	120.6
C(14)-C(13)-C(18)	119.7(2)	C(20)-C(25)-C(12)	113.90(19)
C(18)-C(13)-C(12)	114.6(2)	C(20)-C(25)-C(24)	120.3(2)
C(13)-C(14)-H(14)	120.3	C(24)-C(25)-C(12)	125.4(2)
C(13)-C(14)-C(15)	119.5(2)	C(12)-C(26)-H(26)	110.4
C(15)-C(14)-H(14)	120.3	C(27)-C(26)-C(12)	109.22(18)
C(14)-C(15)-H(15)	120.0	C(27)-C(26)-H(26)	110.4
C(16)-C(15)-C(14)	120.1(2)	C(31)-C(26)-C(12)	112.21(19)
C(16)-C(15)-H(15)	120.0	C(31)-C(26)-H(26)	110.4
C(15)-C(16)-H(16)	119.6	C(31)-C(26)-C(27)	104.10(19)

C(19)-C(27)-H(27)	110.6	C(38)-C(33)-N(30)	120.5(2)
C(26)-C(27)-C(19)	110.90(19)	C(33)-C(34)-H(34)	120.5
C(26)-C(27)-H(27)	110.6	C(33)-C(34)-C(35)	119.1(2)
C(28)-C(27)-C(19)	108.31(19)	C(35)-C(34)-H(34)	120.5
C(28)-C(27)-C(26)	105.61(19)	C(34)-C(35)-H(35)	119.7
C(28)-C(27)-H(27)	110.6	C(34)-C(35)-C(36)	120.7(3)
O(29)-C(28)-C(27)	127.1(2)	C(36)-C(35)-H(35)	119.7
O(29)-C(28)-N(30)	124.4(2)	C(35)-C(36)-H(36)	120.3
N(30)-C(28)-C(27)	108.5(2)	C(37)-C(36)-C(35)	119.4(3)
C(28)-N(30)-C(33)	122.2(2)	C(37)-C(36)-H(36)	120.3
C(31)-N(30)-C(28)	113.1(2)	C(36)-C(37)-H(37)	119.8
C(31)-N(30)-C(33)	124.7(2)	C(36)-C(37)-C(38)	120.5(3)
N(30)-C(31)-C(26)	108.5(2)	C(38)-C(37)-H(37)	119.8
O(32)-C(31)-C(26)	127.8(2)	C(33)-C(38)-H(38)	120.3
O(32)-C(31)-N(30)	123.7(2)	C(37)-C(38)-C(33)	119.3(3)
C(34)-C(33)-N(30)	118.5(2)	C(37)-C(38)-H(38)	120.3
C(34)-C(33)-C(38)	121.0(2)		

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd813. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	16(1)	21(1)	16(1)	-2(1)	4(1)	1(1)
C(2)	19(1)	16(1)	14(1)	-2(1)	5(1)	1(1)
C(3)	21(1)	16(1)	16(1)	2(1)	5(1)	-2(1)
C(4)	20(1)	15(1)	16(1)	3(1)	2(1)	-2(1)
C(5)	18(1)	14(1)	14(1)	0(1)	3(1)	2(1)
C(6)	20(1)	14(1)	17(1)	4(1)	7(1)	3(1)
C(7)	20(1)	16(1)	15(1)	4(1)	5(1)	4(1)
C(8)	18(1)	16(1)	18(1)	2(1)	4(1)	1(1)
O(9)	20(1)	22(1)	36(1)	10(1)	3(1)	-1(1)
C(10)	17(1)	15(1)	16(1)	0(1)	4(1)	0(1)
C(11)	17(1)	14(1)	17(1)	2(1)	5(1)	0(1)
C(12)	12(1)	13(1)	13(1)	0(1)	2(1)	-1(1)
C(13)	16(1)	13(1)	15(1)	-2(1)	5(1)	-3(1)
C(14)	22(1)	17(1)	14(1)	-1(1)	5(1)	-3(1)
C(15)	28(1)	18(1)	18(1)	-4(1)	12(1)	-7(1)
C(16)	19(1)	17(1)	25(1)	-6(1)	12(1)	-5(1)
C(17)	16(1)	13(1)	21(1)	-3(1)	7(1)	-2(1)
C(18)	16(1)	11(1)	14(1)	-3(1)	5(1)	-3(1)
C(19)	13(1)	11(1)	16(1)	-1(1)	4(1)	2(1)
C(20)	13(1)	10(1)	14(1)	-2(1)	3(1)	-3(1)
C(21)	18(1)	12(1)	16(1)	0(1)	3(1)	0(1)
C(22)	26(1)	17(1)	14(1)	0(1)	6(1)	-2(1)
C(23)	23(1)	15(1)	18(1)	-2(1)	9(1)	1(1)
C(24)	17(1)	10(1)	16(1)	0(1)	5(1)	2(1)
C(25)	13(1)	9(1)	13(1)	1(1)	5(1)	-2(1)
C(26)	14(1)	14(1)	15(1)	-1(1)	3(1)	-2(1)
C(27)	15(1)	11(1)	17(1)	-1(1)	5(1)	0(1)
C(28)	13(1)	12(1)	23(1)	-4(1)	5(1)	-3(1)
O(29)	16(1)	12(1)	34(1)	3(1)	6(1)	1(1)
N(30)	13(1)	11(1)	18(1)	1(1)	4(1)	-2(1)
C(31)	14(1)	13(1)	16(1)	0(1)	1(1)	-1(1)
O(32)	14(1)	20(1)	29(1)	6(1)	3(1)	0(1)
C(33)	15(1)	12(1)	21(1)	4(1)	4(1)	-1(1)

C(34)	21(1)	20(1)	22(1)	3(1)	4(1)	-4(1)
C(35)	28(1)	27(1)	25(1)	8(1)	10(1)	-2(1)
C(36)	25(1)	28(2)	38(2)	13(1)	13(1)	-2(1)
C(37)	19(1)	28(2)	39(2)	8(1)	5(1)	-7(1)
C(38)	16(1)	19(1)	24(1)	2(1)	2(1)	-2(1)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd813.

	x	y	z	U(eq)
H(3)	422	6164	5324	21
H(4)	2188	6031	5633	21
H(6)	2368	9751	4503	20
H(7)	606	9772	4155	21
H(10)	3759	5195	5396	19
H(11)	5114	7356	6124	19
H(14)	6343	7048	5566	21
H(15)	7988	7161	5458	24
H(16)	9234	5360	6000	23
H(17)	8841	3344	6621	20
H(19)	7504	1907	7018	16
H(21)	7120	2516	7979	19
H(22)	6150	3937	8491	23
H(23)	4890	5818	8021	22
H(24)	4616	6368	7025	17
H(26)	5111	2909	5615	17
H(27)	6361	1131	6060	18
H(34)	4885	859	7726	25
H(35)	3885	-452	8249	31
H(36)	2383	-1808	7761	35
H(37)	1887	-1858	6746	35
H(38)	2891	-581	6219	25

Table 7. Torsion angles [°] for tcd813.

I(1)-C(2)-C(3)-C(4)	172.62(19)	C(13)-C(12)-C(26)-C(31)	168.52(19)
I(1)-C(2)-C(7)-C(6)	-174.68(18)	C(13)-C(14)-C(15)-C(16)	-1.4(4)
C(2)-C(3)-C(4)-C(5)	3.2(4)	C(13)-C(18)-C(19)-C(20)	-54.6(2)
C(3)-C(2)-C(7)-C(6)	2.0(4)	C(13)-C(18)-C(19)-C(27)	57.8(2)
C(3)-C(4)-C(5)-C(6)	-0.4(4)	C(14)-C(13)-C(18)-C(17)	2.6(3)
C(3)-C(4)-C(5)-C(8)	178.2(2)	C(14)-C(13)-C(18)-C(19)	-176.2(2)
C(4)-C(5)-C(6)-C(7)	-1.7(4)	C(14)-C(15)-C(16)-C(17)	1.7(4)
C(4)-C(5)-C(8)-O(9)	-149.6(3)	C(15)-C(16)-C(17)-C(18)	0.1(4)
C(4)-C(5)-C(8)-C(10)	29.5(3)	C(16)-C(17)-C(18)-C(13)	-2.3(3)
C(5)-C(6)-C(7)-C(2)	0.9(4)	C(16)-C(17)-C(18)-C(19)	176.3(2)
C(5)-C(8)-C(10)-C(11)	-147.8(2)	C(17)-C(18)-C(19)-C(20)	126.7(2)
C(6)-C(5)-C(8)-O(9)	28.9(4)	C(17)-C(18)-C(19)-C(27)	-120.9(2)
C(6)-C(5)-C(8)-C(10)	-152.0(2)	C(18)-C(13)-C(14)-C(15)	-0.7(3)
C(7)-C(2)-C(3)-C(4)	-4.0(4)	C(18)-C(19)-C(20)-C(21)	-132.7(2)
C(8)-C(5)-C(6)-C(7)	179.8(2)	C(18)-C(19)-C(20)-C(25)	53.1(3)
C(8)-C(10)-C(11)-C(12)	-175.2(2)	C(18)-C(19)-C(27)-C(26)	-60.0(2)
O(9)-C(8)-C(10)-C(11)	31.3(4)	C(18)-C(19)-C(27)-C(28)	-175.42(19)
C(10)-C(11)-C(12)-C(13)	117.3(3)	C(19)-C(20)-C(21)-C(22)	-173.9(2)
C(10)-C(11)-C(12)-C(25)	-123.2(3)	C(19)-C(20)-C(25)-C(12)	0.4(3)
C(10)-C(11)-C(12)-C(26)	-2.0(4)	C(19)-C(20)-C(25)-C(24)	173.5(2)
C(11)-C(12)-C(13)-C(14)	-8.7(3)	C(19)-C(27)-C(28)-O(29)	-62.9(3)
C(11)-C(12)-C(13)-C(18)	172.21(19)	C(19)-C(27)-C(28)-N(30)	115.5(2)
C(11)-C(12)-C(25)-C(20)	-171.02(19)	C(20)-C(19)-C(27)-C(26)	54.4(2)
C(11)-C(12)-C(25)-C(24)	16.2(3)	C(20)-C(19)-C(27)-C(28)	-61.1(2)
C(11)-C(12)-C(26)-C(27)	174.63(19)	C(20)-C(21)-C(22)-C(23)	1.1(4)
C(11)-C(12)-C(26)-C(31)	-70.5(3)	C(21)-C(20)-C(25)-C(12)	-174.1(2)
C(12)-C(13)-C(14)-C(15)	-179.7(2)	C(21)-C(20)-C(25)-C(24)	-0.9(3)
C(12)-C(13)-C(18)-C(17)	-178.3(2)	C(21)-C(22)-C(23)-C(24)	-1.0(4)
C(12)-C(13)-C(18)-C(19)	2.9(3)	C(22)-C(23)-C(24)-C(25)	0.0(4)
C(12)-C(26)-C(27)-C(19)	3.8(3)	C(23)-C(24)-C(25)-C(12)	173.3(2)
C(12)-C(26)-C(27)-C(28)	120.9(2)	C(23)-C(24)-C(25)-C(20)	1.0(3)
C(12)-C(26)-C(31)-N(30)	-116.1(2)	C(25)-C(12)-C(13)-C(14)	-131.4(2)
C(12)-C(26)-C(31)-O(32)	62.6(3)	C(25)-C(12)-C(13)-C(18)	49.6(3)
C(13)-C(12)-C(25)-C(20)	-51.2(3)	C(25)-C(12)-C(26)-C(27)	-59.5(2)
C(13)-C(12)-C(25)-C(24)	136.1(2)	C(25)-C(12)-C(26)-C(31)	55.4(2)
C(13)-C(12)-C(26)-C(27)	53.6(2)	C(25)-C(20)-C(21)-C(22)	-0.1(4)

C(26)-C(12)-C(13)-C(14)	118.3(2)	O(29)-C(28)-N(30)-C(31)	-176.7(2)
C(26)-C(12)-C(13)-C(18)	-60.8(2)	O(29)-C(28)-N(30)-C(33)	5.6(4)
C(26)-C(12)-C(25)-C(20)	60.0(2)	N(30)-C(33)-C(34)-C(35)	-179.0(2)
C(26)-C(12)-C(25)-C(24)	-112.7(2)	N(30)-C(33)-C(38)-C(37)	179.4(2)
C(26)-C(27)-C(28)-O(29)	178.3(2)	C(31)-C(26)-C(27)-C(19)	-116.2(2)
C(26)-C(27)-C(28)-N(30)	-3.4(2)	C(31)-C(26)-C(27)-C(28)	0.9(2)
C(27)-C(19)-C(20)-C(21)	115.1(2)	C(31)-N(30)-C(33)-C(34)	-117.6(3)
C(27)-C(19)-C(20)-C(25)	-59.1(2)	C(31)-N(30)-C(33)-C(38)	63.3(3)
C(27)-C(26)-C(31)-N(30)	1.9(2)	C(33)-N(30)-C(31)-C(26)	173.3(2)
C(27)-C(26)-C(31)-O(32)	-179.5(3)	C(33)-N(30)-C(31)-O(32)	-5.4(4)
C(27)-C(28)-N(30)-C(31)	4.9(3)	C(33)-C(34)-C(35)-C(36)	-0.4(4)
C(27)-C(28)-N(30)-C(33)	-172.8(2)	C(34)-C(33)-C(38)-C(37)	0.3(4)
C(28)-N(30)-C(31)-C(26)	-4.3(3)	C(34)-C(35)-C(36)-C(37)	0.2(5)
C(28)-N(30)-C(31)-O(32)	177.0(2)	C(35)-C(36)-C(37)-C(38)	0.3(5)
C(28)-N(30)-C(33)-C(34)	59.8(3)	C(36)-C(37)-C(38)-C(33)	-0.5(4)
C(28)-N(30)-C(33)-C(38)	-119.3(3)	C(38)-C(33)-C(34)-C(35)	0.1(4)

Table 8. Hydrogen bonds for tcd813 [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(38)-H(38)...I(1)#1	0.95	3.20	3.668(3)	113

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1



Small Molecule X-ray Facility School Of Chemistry

Trinity College Dublin
The University of Dublin

Structure Report

Filename: TCD1371

Submitted by: James McKeown

Reference: KAI C

Group: Meegan

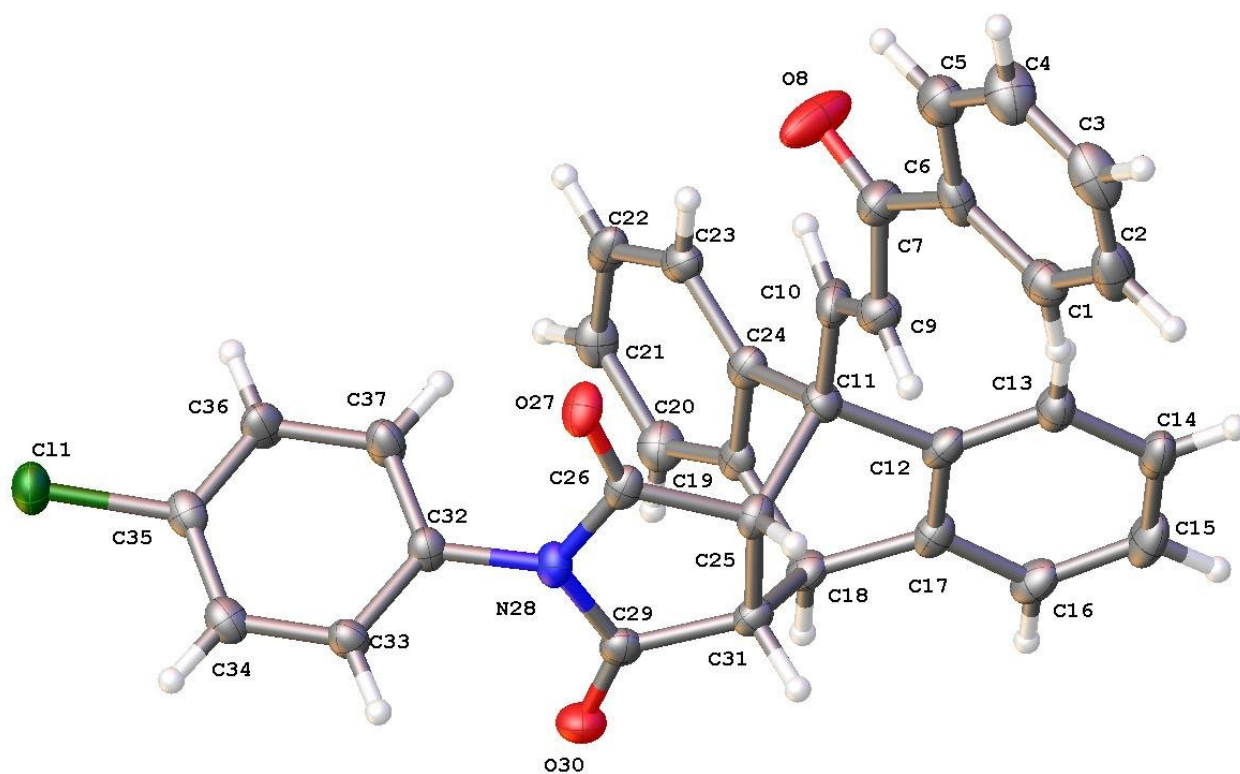


Fig. 1. Molecular structure of TCD1371, with atomic displacement shown at 50% probability.

2/10/19

Author: Brendan Twamley

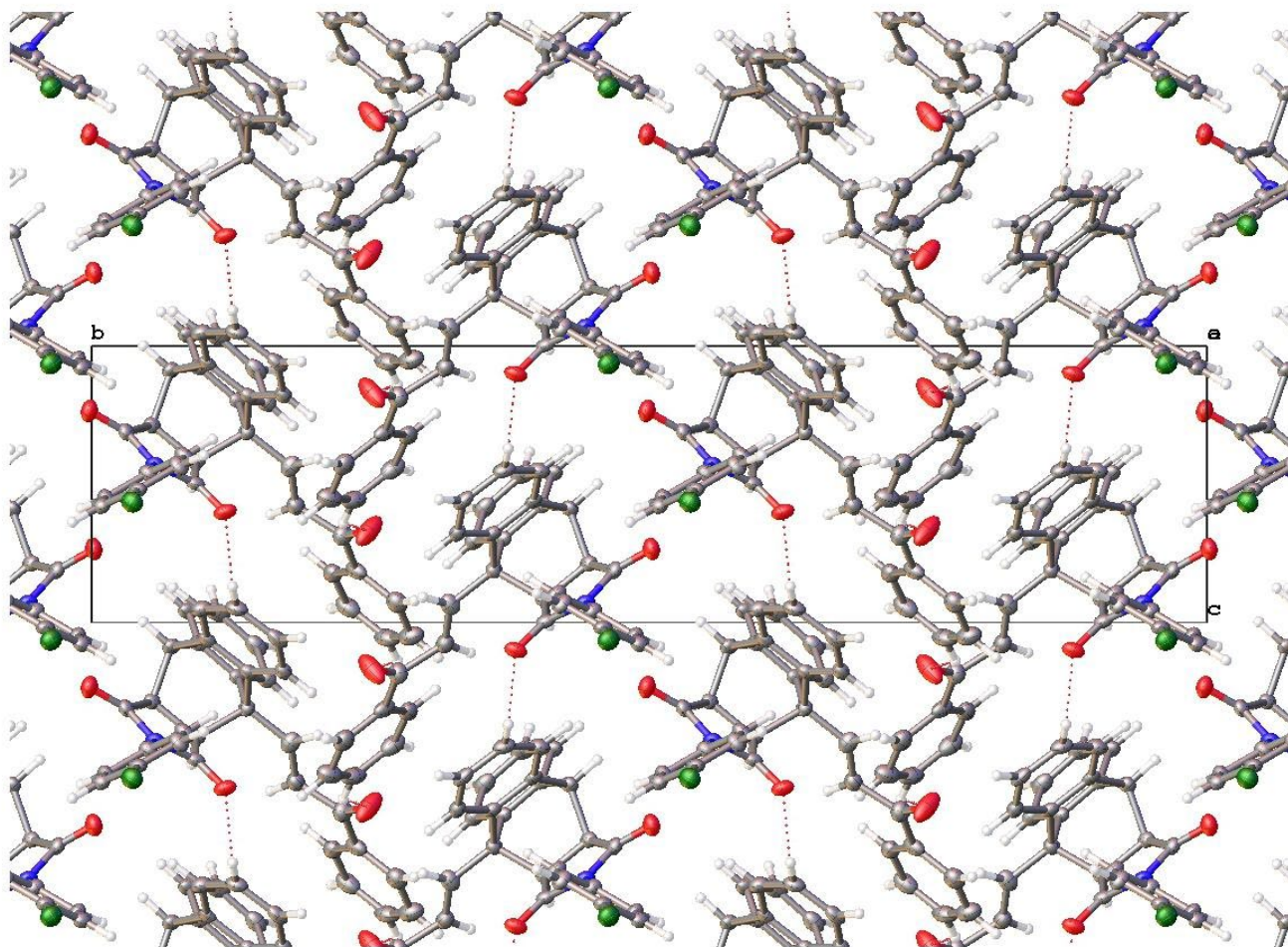


Fig. 2. Packing diagram of TCD1371 viewed normal to the a-axis. Dotted lines indicate weak hydrogen bonding interactions. Displacement shown at 50% probability.

Crystal Structure Report for TCD1371

A specimen of $\text{C}_{33}\text{H}_{22}\text{ClNO}_3$, approximate dimensions 0.050 mm x 0.110 mm x 0.150 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured ($\lambda = 1.54178$ Å) at 100(2)K on a Bruker Apex Kappa Duo with an Oxford Cobra Cryosystem low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 2333 frames were collected. The total exposure time was 40.17 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a *monoclinic* unit cell yielded a total of 35493 reflections to a maximum θ angle of 68.45° (0.83 Å resolution), of which 3970 were independent (average redundancy 8.940, completeness = 99.7%, $R_{\text{int}} = 9.69\%$, $R_{\text{sig}} = 4.69\%$) and 3506 (88.31%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 11.0462(5)$ Å, $b = 30.0538(14)$ Å, $c = 8.1065(4)$ Å, $\beta = 113.531(3)^\circ$, volume = $2467.4(2)$ Å³, are based upon the refinement of the XYZ-centroids of 5333 reflections above $20\sigma(I)$ with $5.881^\circ < 2\theta < 136.4^\circ$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.955. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7880 and 0.9210.

The structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimisation with Olex2, using the space group *Cc*, with $Z = 4$ for the formula unit, $\text{C}_{33}\text{H}_{22}\text{ClNO}_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 343 variables converged at $R1 = 6.54\%$, for the observed data and $wR2 = 18.44\%$ for all data. The goodness-of-fit was 1.104. The largest peak in the final difference electron density synthesis was $0.557 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.296 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.078 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.389 g/cm^3 and $F(000)$, 1072 e^- .

Refinement Note: Poorly diffracting small crystal. Weak high angle data and incomplete Friedel pair collection. Model has chirality at C25 and C31, S (Polar Space Group).

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Table 1: Data collection details for TCD1371.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	50.046	108.90	95.60	240.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	24.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	96.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	190.25	64.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	192.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	190.25	288.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	99.93	352.54	294.44	82.95	1.40	52	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	190.26	360.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	108.90	342.80	240.00	64.50	1.40	99	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	298.82	192.00	-64.50	1.40	83	35.00	1.54184	50	0.6	100
Omega	50.046	108.90	342.80	144.00	64.50	1.40	99	80.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	264.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	168.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	-11.30	228.26	153.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	48.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	190.25	320.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	108.90	342.80	24.00	64.50	1.40	99	80.00	1.54184	50	0.6	100
Omega	50.046	98.68	353.31	97.12	80.65	1.40	52	80.00	1.54184	50	0.6	100
Omega	50.046	108.90	342.80	312.00	64.50	1.40	99	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	298.82	96.00	-64.50	1.40	83	35.00	1.54184	50	0.6	100
Phi	50.046	109.30	4.12	4.00	23.00	1.40	131	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	190.26	128.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	108.90	95.60	72.00	-54.74	1.40	96	80.00	1.54184	50	0.6	100
Omega	50.046	-49.30	190.25	192.00	54.74	1.40	96	35.00	1.54184	50	0.6	100
Omega	50.046	-11.30	228.25	255.00	54.74	1.40	96	35.00	1.54184	50	0.6	100

Table 2. Crystal data and structure refinement for tcd1371.

Identification code	tcd1371	
Empirical formula	C ₃₃ H ₂₂ ClNO ₃	
Formula weight	515.96	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 11.0462(5) Å	$\alpha = 90^\circ$.
	b = 30.0538(14) Å	$\beta = 113.531(3)^\circ$.
	c = 8.1065(4) Å	$\gamma = 90^\circ$.
Volume	2467.4(2) Å ³	
Z	4	
Density (calculated)	1.389 Mg/m ³	
Absorption coefficient	1.671 mm ⁻¹	
F(000)	1072	
Crystal size	0.15 x 0.11 x 0.05 mm ³	
Theta range for data collection	2.941 to 68.453°.	
Index ranges	-11 ≤ h ≤ 13, -36 ≤ k ≤ 36, -9 ≤ l ≤ 9	
Reflections collected	35493	
Independent reflections	3970 [R(int) = 0.0969]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7350 and 0.7022	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3970 / 2 / 343	
Goodness-of-fit on F ²	1.104	
Final R indices [I > 2σ(I)]	R1 = 0.0654, wR2 = 0.1758	
R indices (all data)	R1 = 0.0740, wR2 = 0.1844	
Absolute structure parameter	-0.04(2)	
Largest diff. peak and hole	0.557 and -0.296 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd1371. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
Cl(1)	11095(2)	4637(1)	5638(2)	31(1)
O(8)	4227(6)	2513(2)	6688(9)	55(2)
O(27)	5772(4)	3803(2)	6000(6)	28(1)
O(30)	4540(5)	5020(1)	2337(7)	32(1)
N(28)	5453(5)	4430(2)	4274(7)	22(1)
C(1)	1691(7)	2840(2)	8219(9)	30(1)
C(2)	1186(7)	2699(3)	9440(10)	38(2)
C(3)	1749(8)	2349(3)	10554(9)	41(2)
C(4)	2803(8)	2134(3)	10481(10)	41(2)
C(5)	3329(7)	2262(2)	9273(10)	36(2)
C(6)	2779(7)	2625(2)	8126(9)	30(1)
C(7)	3391(7)	2748(2)	6840(9)	31(1)
C(9)	2989(6)	3169(2)	5769(9)	28(1)
C(10)	3291(6)	3214(2)	4343(9)	28(1)
C(11)	3003(6)	3609(2)	3100(8)	23(1)
C(12)	1517(6)	3683(2)	1970(8)	24(1)
C(13)	496(6)	3430(2)	2040(9)	27(1)
C(14)	-802(6)	3526(2)	845(9)	31(1)
C(15)	-1065(7)	3869(3)	-368(9)	34(2)
C(16)	-31(6)	4133(2)	-418(8)	29(1)
C(17)	1248(6)	4034(2)	764(8)	24(1)
C(18)	2458(6)	4286(2)	870(8)	23(1)
C(19)	3414(6)	3961(2)	627(7)	22(1)
C(20)	4071(7)	4016(2)	-501(8)	29(1)
C(21)	4979(6)	3700(2)	-521(8)	28(1)
C(22)	5234(6)	3335(2)	583(9)	28(1)
C(23)	4602(6)	3279(2)	1761(8)	25(1)
C(24)	3683(6)	3592(2)	1780(8)	23(1)
C(25)	3524(6)	4062(2)	4159(8)	22(1)
C(26)	5028(6)	4067(2)	4960(7)	21(1)
C(29)	4424(7)	4689(2)	3101(8)	23(1)
C(31)	3135(6)	4458(2)	2860(8)	22(1)
C(32)	6813(6)	4492(2)	4585(8)	23(1)
C(33)	7494(6)	4872(2)	5477(8)	24(1)
C(34)	8809(6)	4917(2)	5771(8)	26(1)

C(35)	9405(6)	4588(2)	5193(8)	24(1)
C(36)	8752(7)	4216(2)	4322(9)	28(1)
C(37)	7423(7)	4168(2)	3992(9)	28(1)

Table 4. Bond lengths [Å] and angles [°] for tcd1371.

Cl(1)-C(35)	1.761(7)	C(18)-H(18)	1.0000
O(8)-C(7)	1.207(8)	C(18)-C(19)	1.508(8)
O(27)-C(26)	1.210(7)	C(18)-C(31)	1.570(8)
O(30)-C(29)	1.206(7)	C(19)-C(20)	1.386(9)
N(28)-C(26)	1.389(8)	C(19)-C(24)	1.403(8)
N(28)-C(29)	1.391(8)	C(20)-H(20)	0.9500
N(28)-C(32)	1.432(8)	C(20)-C(21)	1.387(9)
C(1)-H(1)	0.9500	C(21)-H(21)	0.9500
C(1)-C(2)	1.381(10)	C(21)-C(22)	1.372(10)
C(1)-C(6)	1.393(10)	C(22)-H(22)	0.9500
C(2)-H(2)	0.9500	C(22)-C(23)	1.400(9)
C(2)-C(3)	1.364(12)	C(23)-H(23)	0.9500
C(3)-H(3)	0.9500	C(23)-C(24)	1.389(8)
C(3)-C(4)	1.355(12)	C(25)-H(25)	1.0000
C(4)-H(4)	0.9500	C(25)-C(26)	1.523(8)
C(4)-C(5)	1.378(10)	C(25)-C(31)	1.531(8)
C(5)-H(5)	0.9500	C(29)-C(31)	1.525(8)
C(5)-C(6)	1.405(10)	C(31)-H(31)	1.0000
C(6)-C(7)	1.498(9)	C(32)-C(33)	1.398(8)
C(7)-C(9)	1.497(9)	C(32)-C(37)	1.377(9)
C(9)-H(9)	0.9500	C(33)-H(33)	0.9500
C(9)-C(10)	1.332(9)	C(33)-C(34)	1.381(9)
C(10)-H(10)	0.9500	C(34)-H(34)	0.9500
C(10)-C(11)	1.505(8)	C(34)-C(35)	1.370(9)
C(11)-C(12)	1.543(9)	C(35)-C(36)	1.363(9)
C(11)-C(24)	1.534(8)	C(36)-H(36)	0.9500
C(11)-C(25)	1.591(8)	C(36)-C(37)	1.391(9)
C(12)-C(13)	1.380(9)	C(37)-H(37)	0.9500
C(12)-C(17)	1.388(9)		
C(13)-H(13)	0.9500	C(26)-N(28)-C(29)	113.5(5)
C(13)-C(14)	1.403(9)	C(26)-N(28)-C(32)	122.0(5)
C(14)-H(14)	0.9500	C(29)-N(28)-C(32)	124.1(5)
C(14)-C(15)	1.374(10)	C(2)-C(1)-H(1)	119.9
C(15)-H(15)	0.9500	C(2)-C(1)-C(6)	120.1(6)
C(15)-C(16)	1.403(10)	C(6)-C(1)-H(1)	119.9
C(16)-H(16)	0.9500	C(1)-C(2)-H(2)	119.9
C(16)-C(17)	1.386(9)	C(3)-C(2)-C(1)	120.2(7)
C(17)-C(18)	1.507(9)	C(3)-C(2)-H(2)	119.9

C(2)-C(3)-H(3)	119.6	C(17)-C(16)-C(15)	118.5(6)
C(4)-C(3)-C(2)	120.8(6)	C(17)-C(16)-H(16)	120.8
C(4)-C(3)-H(3)	119.6	C(12)-C(17)-C(18)	113.9(5)
C(3)-C(4)-H(4)	119.6	C(16)-C(17)-C(12)	121.4(6)
C(3)-C(4)-C(5)	120.8(7)	C(16)-C(17)-C(18)	124.7(6)
C(5)-C(4)-H(4)	119.6	C(17)-C(18)-H(18)	112.0
C(4)-C(5)-H(5)	120.2	C(17)-C(18)-C(19)	108.6(5)
C(4)-C(5)-C(6)	119.6(7)	C(17)-C(18)-C(31)	105.8(5)
C(6)-C(5)-H(5)	120.2	C(19)-C(18)-H(18)	112.0
C(1)-C(6)-C(5)	118.6(6)	C(19)-C(18)-C(31)	106.0(5)
C(1)-C(6)-C(7)	124.3(6)	C(31)-C(18)-H(18)	112.0
C(5)-C(6)-C(7)	117.1(6)	C(20)-C(19)-C(18)	125.9(6)
O(8)-C(7)-C(6)	120.2(6)	C(20)-C(19)-C(24)	120.4(6)
O(8)-C(7)-C(9)	119.8(6)	C(24)-C(19)-C(18)	113.5(5)
C(9)-C(7)-C(6)	120.0(6)	C(19)-C(20)-H(20)	120.1
C(7)-C(9)-H(9)	120.9	C(19)-C(20)-C(21)	119.8(6)
C(10)-C(9)-C(7)	118.1(6)	C(21)-C(20)-H(20)	120.1
C(10)-C(9)-H(9)	120.9	C(20)-C(21)-H(21)	120.0
C(9)-C(10)-H(10)	116.5	C(22)-C(21)-C(20)	120.1(6)
C(9)-C(10)-C(11)	127.1(6)	C(22)-C(21)-H(21)	120.0
C(11)-C(10)-H(10)	116.5	C(21)-C(22)-H(22)	119.6
C(10)-C(11)-C(12)	113.7(5)	C(21)-C(22)-C(23)	120.8(6)
C(10)-C(11)-C(24)	114.2(5)	C(23)-C(22)-H(22)	119.6
C(10)-C(11)-C(25)	112.3(5)	C(22)-C(23)-H(23)	120.2
C(12)-C(11)-C(25)	105.4(5)	C(24)-C(23)-C(22)	119.5(6)
C(24)-C(11)-C(12)	106.9(5)	C(24)-C(23)-H(23)	120.2
C(24)-C(11)-C(25)	103.4(4)	C(19)-C(24)-C(11)	114.0(5)
C(13)-C(12)-C(11)	126.1(5)	C(23)-C(24)-C(11)	126.4(5)
C(13)-C(12)-C(17)	120.0(6)	C(23)-C(24)-C(19)	119.3(5)
C(17)-C(12)-C(11)	114.0(6)	C(11)-C(25)-H(25)	110.7
C(12)-C(13)-H(13)	120.5	C(26)-C(25)-C(11)	109.7(5)
C(12)-C(13)-C(14)	119.0(6)	C(26)-C(25)-H(25)	110.7
C(14)-C(13)-H(13)	120.5	C(26)-C(25)-C(31)	104.1(5)
C(13)-C(14)-H(14)	119.5	C(31)-C(25)-C(11)	110.6(5)
C(15)-C(14)-C(13)	121.0(6)	C(31)-C(25)-H(25)	110.7
C(15)-C(14)-H(14)	119.5	O(27)-C(26)-N(28)	123.5(5)
C(14)-C(15)-H(15)	119.9	O(27)-C(26)-C(25)	127.7(5)
C(14)-C(15)-C(16)	120.1(6)	N(28)-C(26)-C(25)	108.8(5)
C(16)-C(15)-H(15)	119.9	O(30)-C(29)-N(28)	125.9(6)
C(15)-C(16)-H(16)	120.8	O(30)-C(29)-C(31)	126.5(6)

N(28)-C(29)-C(31)	107.4(5)	C(33)-C(34)-H(34)	120.5
C(18)-C(31)-H(31)	110.8	C(35)-C(34)-C(33)	119.1(6)
C(25)-C(31)-C(18)	109.8(5)	C(35)-C(34)-H(34)	120.5
C(25)-C(31)-H(31)	110.8	C(34)-C(35)-Cl(1)	119.0(5)
C(29)-C(31)-C(18)	108.6(5)	C(36)-C(35)-Cl(1)	118.3(5)
C(29)-C(31)-C(25)	105.9(5)	C(36)-C(35)-C(34)	122.7(6)
C(29)-C(31)-H(31)	110.8	C(35)-C(36)-H(36)	120.5
C(33)-C(32)-N(28)	120.5(5)	C(35)-C(36)-C(37)	119.0(6)
C(37)-C(32)-N(28)	118.2(5)	C(37)-C(36)-H(36)	120.5
C(37)-C(32)-C(33)	121.4(6)	C(32)-C(37)-C(36)	119.0(6)
C(32)-C(33)-H(33)	120.6	C(32)-C(37)-H(37)	120.5
C(34)-C(33)-C(32)	118.8(6)	C(36)-C(37)-H(37)	120.5
C(34)-C(33)-H(33)	120.6		

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd1371. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
Cl(1)	23(1)	37(1)	35(1)	-3(1)	12(1)	-5(1)
O(8)	63(4)	55(3)	71(4)	33(3)	52(3)	34(3)
O(27)	20(2)	41(2)	20(2)	7(2)	5(2)	1(2)
O(30)	34(3)	22(2)	39(3)	4(2)	13(2)	3(2)
N(28)	18(2)	28(2)	18(2)	-2(2)	5(2)	-3(2)
C(1)	29(4)	29(3)	28(3)	-2(2)	6(3)	-5(3)
C(2)	32(4)	49(4)	38(4)	-16(3)	20(3)	-12(3)
C(3)	47(4)	53(4)	25(4)	-4(3)	16(3)	-18(4)
C(4)	38(4)	52(4)	30(3)	6(3)	12(3)	-7(3)
C(5)	31(4)	37(3)	36(4)	-6(3)	10(3)	-3(3)
C(6)	28(3)	38(3)	26(3)	-10(3)	13(3)	-15(3)
C(7)	28(3)	31(3)	36(4)	1(3)	14(3)	3(3)
C(9)	27(3)	27(3)	28(3)	-2(2)	8(3)	1(2)
C(10)	18(3)	33(3)	35(3)	6(3)	10(3)	0(2)
C(11)	23(3)	23(3)	22(3)	1(2)	9(3)	-2(2)
C(12)	21(3)	28(3)	21(3)	-3(2)	5(2)	7(2)
C(13)	20(3)	32(3)	27(3)	-5(2)	6(3)	-1(2)
C(14)	19(3)	36(3)	36(4)	-12(3)	7(3)	-1(3)
C(15)	19(3)	49(4)	28(3)	-9(3)	1(3)	3(3)
C(16)	25(3)	33(3)	22(3)	-1(2)	2(3)	9(2)
C(17)	21(3)	32(3)	19(3)	-3(2)	7(2)	3(2)
C(18)	20(3)	25(3)	21(3)	3(2)	4(2)	4(2)
C(19)	17(3)	26(3)	17(3)	-7(2)	2(2)	-2(2)
C(20)	29(3)	36(3)	21(3)	3(2)	9(3)	0(3)
C(21)	27(3)	41(3)	20(3)	-5(2)	11(3)	-5(3)
C(22)	21(3)	34(3)	28(3)	-14(3)	8(3)	0(2)
C(23)	22(3)	24(3)	24(3)	-4(2)	4(2)	1(2)
C(24)	20(3)	28(3)	18(3)	-5(2)	5(2)	0(2)
C(25)	18(3)	30(3)	18(3)	1(2)	7(2)	1(2)
C(26)	19(3)	29(3)	16(3)	-4(2)	6(2)	1(2)
C(29)	25(3)	21(3)	25(3)	-4(2)	10(3)	1(2)
C(31)	21(3)	23(3)	21(3)	-1(2)	8(2)	4(2)
C(32)	22(3)	27(3)	21(3)	-4(2)	10(2)	-3(2)
C(33)	26(3)	22(3)	23(3)	-5(2)	10(3)	-3(2)
C(34)	28(3)	27(3)	20(3)	1(2)	9(3)	-5(2)

C(35)	24(3)	29(3)	16(3)	0(2)	5(3)	-5(2)
C(36)	28(3)	30(3)	30(3)	-8(3)	18(3)	-3(2)
C(37)	25(3)	31(3)	30(3)	-11(2)	12(3)	-7(3)

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd1371.

	x	y	z	U(eq)
H(1)	1294	3083	7442	36
H(2)	445	2847	9503	45
H(3)	1397	2256	11392	49
H(4)	3185	1892	11272	49
H(5)	4059	2106	9216	43
H(9)	2532	3397	6097	33
H(10)	3736	2972	4081	34
H(13)	669	3194	2885	33
H(14)	-1509	3351	875	38
H(15)	-1948	3928	-1175	41
H(16)	-205	4373	-1243	35
H(18)	2233	4535	-21	28
H(20)	3899	4270	-1259	35
H(21)	5426	3736	-1301	34
H(22)	5847	3117	549	34
H(23)	4800	3029	2541	30
H(25)	3182	4102	5120	26
H(31)	2538	4665	3145	26
H(33)	7061	5094	5873	28
H(34)	9293	5172	6366	31
H(36)	9198	3994	3945	33
H(37)	6944	3914	3367	34

Table 7. Torsion angles [°] for tcd1371.

Cl(1)-C(35)-C(36)-C(37)	178.7(5)	C(12)-C(11)-C(24)-C(19)	53.4(6)
O(8)-C(7)-C(9)-C(10)	17.8(10)	C(12)-C(11)-C(24)-C(23)	-133.2(6)
O(30)-C(29)-C(31)-C(18)	63.4(7)	C(12)-C(11)-C(25)-C(26)	-166.4(5)
O(30)-C(29)-C(31)-C(25)	-178.7(6)	C(12)-C(11)-C(25)-C(31)	-52.1(6)
N(28)-C(29)-C(31)-C(18)	-112.6(5)	C(12)-C(13)-C(14)-C(15)	-0.8(9)
N(28)-C(29)-C(31)-C(25)	5.3(6)	C(12)-C(17)-C(18)-C(19)	55.2(7)
N(28)-C(32)-C(33)-C(34)	179.2(5)	C(12)-C(17)-C(18)-C(31)	-58.1(6)
N(28)-C(32)-C(37)-C(36)	-178.4(6)	C(13)-C(12)-C(17)-C(16)	-1.6(9)
C(1)-C(2)-C(3)-C(4)	-0.2(11)	C(13)-C(12)-C(17)-C(18)	178.7(5)
C(1)-C(6)-C(7)-O(8)	-170.9(7)	C(13)-C(14)-C(15)-C(16)	-0.5(10)
C(1)-C(6)-C(7)-C(9)	10.6(10)	C(14)-C(15)-C(16)-C(17)	0.7(10)
C(2)-C(1)-C(6)-C(5)	1.1(9)	C(15)-C(16)-C(17)-C(12)	0.4(9)
C(2)-C(1)-C(6)-C(7)	179.3(6)	C(15)-C(16)-C(17)-C(18)	180.0(6)
C(2)-C(3)-C(4)-C(5)	-0.3(12)	C(16)-C(17)-C(18)-C(19)	-124.4(6)
C(3)-C(4)-C(5)-C(6)	1.1(11)	C(16)-C(17)-C(18)-C(31)	122.2(6)
C(4)-C(5)-C(6)-C(1)	-1.5(10)	C(17)-C(12)-C(13)-C(14)	1.8(9)
C(4)-C(5)-C(6)-C(7)	-179.9(6)	C(17)-C(18)-C(19)-C(20)	132.8(6)
C(5)-C(6)-C(7)-O(8)	7.4(10)	C(17)-C(18)-C(19)-C(24)	-51.8(7)
C(5)-C(6)-C(7)-C(9)	-171.2(6)	C(17)-C(18)-C(31)-C(25)	60.7(6)
C(6)-C(1)-C(2)-C(3)	-0.2(10)	C(17)-C(18)-C(31)-C(29)	176.1(5)
C(6)-C(7)-C(9)-C(10)	-163.7(6)	C(18)-C(19)-C(20)-C(21)	176.6(6)
C(7)-C(9)-C(10)-C(11)	-179.0(6)	C(18)-C(19)-C(24)-C(11)	-2.8(7)
C(9)-C(10)-C(11)-C(12)	-66.9(8)	C(18)-C(19)-C(24)-C(23)	-176.6(5)
C(9)-C(10)-C(11)-C(24)	170.1(6)	C(19)-C(18)-C(31)-C(25)	-54.5(6)
C(9)-C(10)-C(11)-C(25)	52.8(9)	C(19)-C(18)-C(31)-C(29)	60.9(6)
C(10)-C(11)-C(12)-C(13)	0.9(8)	C(19)-C(20)-C(21)-C(22)	-0.5(10)
C(10)-C(11)-C(12)-C(17)	-177.3(5)	C(20)-C(19)-C(24)-C(11)	172.9(5)
C(10)-C(11)-C(24)-C(19)	-179.9(5)	C(20)-C(19)-C(24)-C(23)	-0.9(8)
C(10)-C(11)-C(24)-C(23)	-6.5(8)	C(20)-C(21)-C(22)-C(23)	-1.0(10)
C(10)-C(11)-C(25)-C(26)	69.3(6)	C(21)-C(22)-C(23)-C(24)	1.5(9)
C(10)-C(11)-C(25)-C(31)	-176.4(5)	C(22)-C(23)-C(24)-C(11)	-173.5(6)
C(11)-C(12)-C(13)-C(14)	-176.3(5)	C(22)-C(23)-C(24)-C(19)	-0.5(8)
C(11)-C(12)-C(17)-C(16)	176.7(5)	C(24)-C(11)-C(12)-C(13)	127.9(6)
C(11)-C(12)-C(17)-C(18)	-2.9(7)	C(24)-C(11)-C(12)-C(17)	-50.3(6)
C(11)-C(25)-C(26)-O(27)	-58.0(7)	C(24)-C(11)-C(25)-C(26)	-54.3(6)
C(11)-C(25)-C(26)-N(28)	120.8(5)	C(24)-C(11)-C(25)-C(31)	60.0(6)
C(11)-C(25)-C(31)-C(18)	-5.2(6)	C(24)-C(19)-C(20)-C(21)	1.5(9)
C(11)-C(25)-C(31)-C(29)	-122.3(5)	C(25)-C(11)-C(12)-C(13)	-122.5(6)

C(25)-C(11)-C(12)-C(17)	59.3(6)	C(31)-C(18)-C(19)-C(24)	61.4(6)
C(25)-C(11)-C(24)-C(19)	-57.6(6)	C(31)-C(25)-C(26)-O(27)	-176.4(6)
C(25)-C(11)-C(24)-C(23)	115.8(6)	C(31)-C(25)-C(26)-N(28)	2.4(6)
C(26)-N(28)-C(29)-O(30)	179.9(6)	C(32)-N(28)-C(26)-O(27)	7.4(8)
C(26)-N(28)-C(29)-C(31)	-4.0(6)	C(32)-N(28)-C(26)-C(25)	-171.5(5)
C(26)-N(28)-C(32)-C(33)	-119.8(6)	C(32)-N(28)-C(29)-O(30)	-7.8(9)
C(26)-N(28)-C(32)-C(37)	59.8(8)	C(32)-N(28)-C(29)-C(31)	168.3(5)
C(26)-C(25)-C(31)-C(18)	112.6(5)	C(32)-C(33)-C(34)-C(35)	-0.5(9)
C(26)-C(25)-C(31)-C(29)	-4.6(5)	C(33)-C(32)-C(37)-C(36)	1.2(10)
C(29)-N(28)-C(26)-O(27)	179.9(5)	C(33)-C(34)-C(35)-Cl(1)	-177.9(5)
C(29)-N(28)-C(26)-C(25)	1.0(6)	C(33)-C(34)-C(35)-C(36)	0.5(9)
C(29)-N(28)-C(32)-C(33)	68.5(8)	C(34)-C(35)-C(36)-C(37)	0.3(9)
C(29)-N(28)-C(32)-C(37)	-111.9(7)	C(35)-C(36)-C(37)-C(32)	-1.2(10)
C(31)-C(18)-C(19)-C(20)	-114.0(6)	C(37)-C(32)-C(33)-C(34)	-0.4(9)

Table 8. Hydrogen bonds for tcd1371 [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(3)-H(3)...O(8)#1	0.95	2.60	3.289(10)	130
C(21)-H(21)...O(27)#2	0.95	2.38	3.284(7)	160

Symmetry transformations used to generate equivalent atoms:

#1 $x-1/2, -y+1/2, z+1/2$ #2 $x, y, z-1$



Small Molecule X-ray Facility School Of Chemistry

Trinity College Dublin

The University of Dublin

Structure Report

Filename: TCD1117

Submitted by: James McKeown

Reference: CC151

Group: Meegan

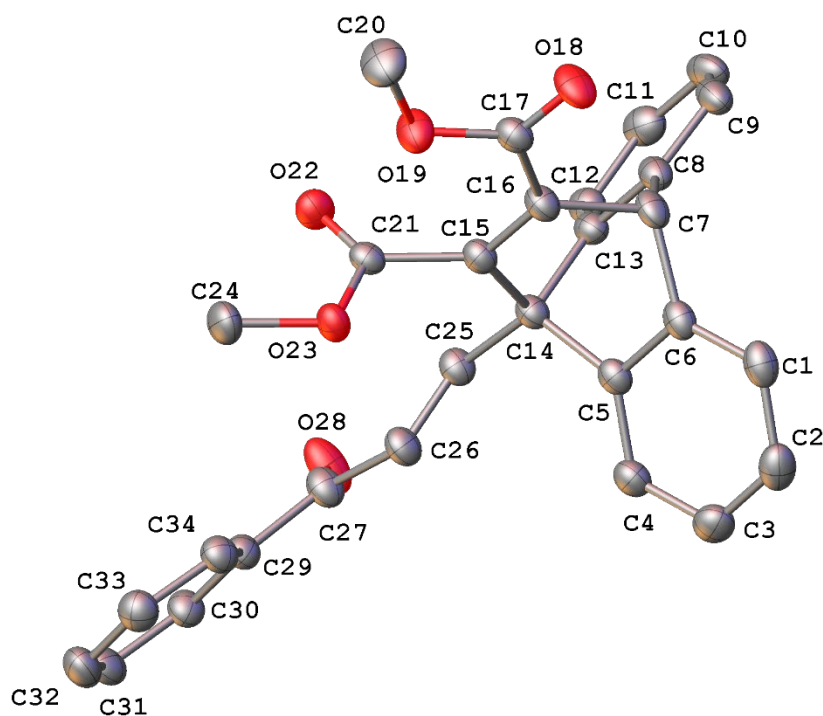


Fig. 1. Labelling scheme in TCD1117 with atomic displacement shown at 50% probability. Hydrogen atoms omitted for clarity.

7/8/18

Author: Brendan Twamley

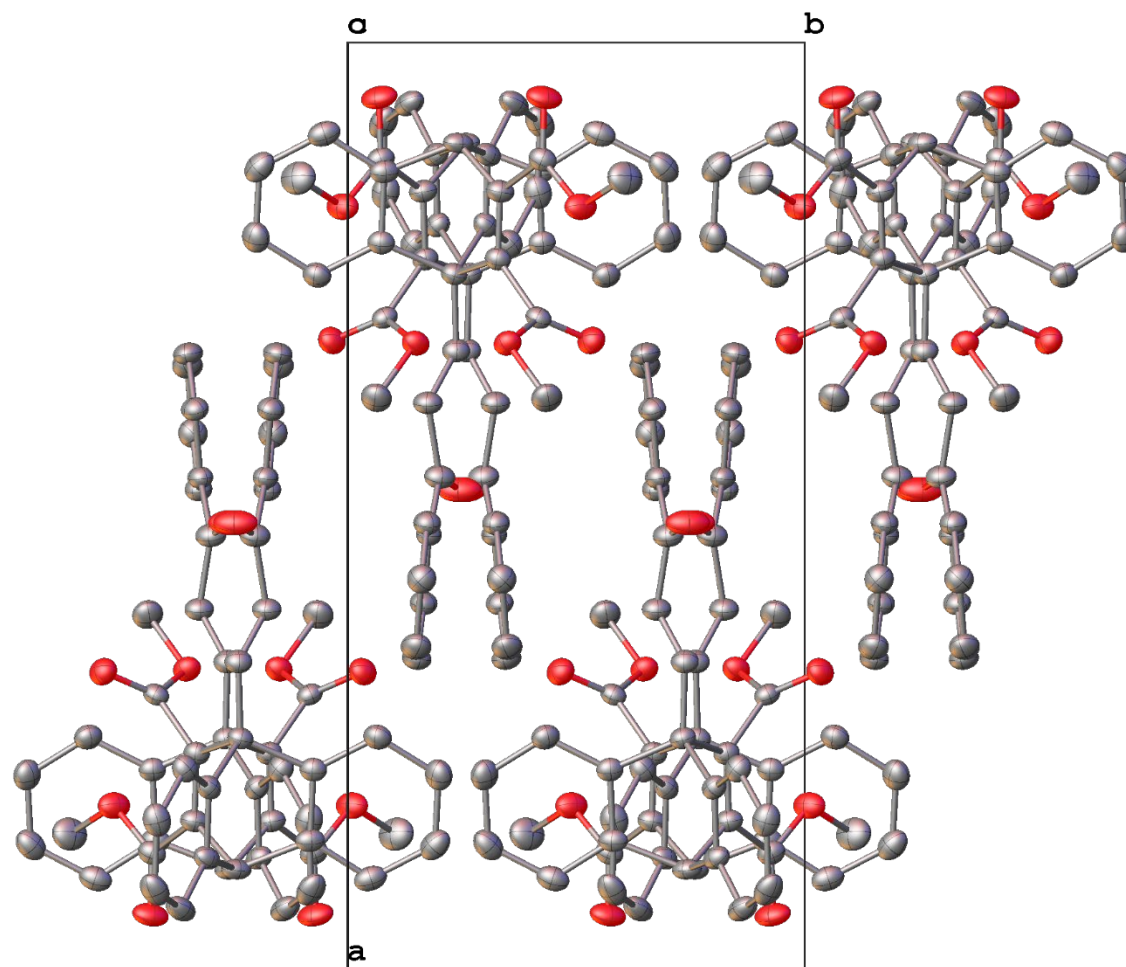


Fig. 2. Packing diagram of TCD1117 viewed normal to the c-axis. Displacement shown at 50% probability. Hydrogen atoms omitted for clarity.

Crystal Structure Report for TCD1117

A specimen of $\text{C}_{29}\text{H}_{22}\text{O}_5$, approximate dimensions 0.140 mm x 0.170 mm x 0.360 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K on a Bruker Apex Kappa Duo with an Oxford Cobra Cryosystem low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 4018 frames were collected. The total exposure time was 13.66 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 27728 reflections to a maximum θ angle of 70.21° (0.82 Å resolution), of which 4310 were independent (average redundancy 6.433, completeness = 99.2%, $R_{\text{int}} = 4.38\%$, $R_{\text{sig}} = 2.67\%$) and 3999 (92.78%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 18.9744(16)$ Å, $b = 8.7405(7)$ Å, $c = 14.7391(12)$ Å, $\beta = 111.108(3)^\circ$, volume = $2280.4(3)$ Å³, are based upon the refinement of the XYZ-centroids of 9105 reflections above $20\sigma(I)$ with $9.994^\circ < 2\theta < 140.3^\circ$. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.842. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7800 and 0.9050.

The structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimisation with Olex2, using the space group $P2_1/c$, with $Z = 4$ for the formula unit, $\text{C}_{29}\text{H}_{22}\text{O}_5$. The final anisotropic full-matrix least-squares refinement on F^2 with 310 variables converged at $R1 = 3.73\%$, for the observed data and $wR2 = 10.27\%$ for all data. The goodness-of-fit was 1.057. The largest peak in the final difference electron density synthesis was $0.218 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.215 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.041 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.312 g/cm^3 and $F(000)$, 944 e^- .

References:

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Table 1: Data collection details for TCD1117.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	50.011	108.90	95.00	168.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	24.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	216.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	-49.30	189.66	64.00	54.74	1.00	135	8.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	120.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Phi	50.011	-47.74	343.92	260.00	23.00	1.00	184	8.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	72.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	-49.30	189.65	0.00	54.74	1.00	135	8.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	0.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	144.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	192.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	-49.30	189.65	320.00	54.74	1.00	135	8.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	48.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	-49.30	299.02	64.00	-64.50	1.00	116	8.00	1.54184	50	0.6	100
Omega	50.011	-11.30	227.66	51.00	54.74	1.00	135	8.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	96.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Omega	50.011	108.90	95.00	336.00	-54.74	1.00	135	15.00	1.54184	50	0.6	100
Phi	50.011	79.30	65.73	0.00	-57.00	1.00	360	15.00	1.54184	50	0.6	100
Omega	50.011	-49.30	189.65	160.00	54.74	1.00	135	8.00	1.54184	50	0.6	100
Omega	50.011	-49.30	299.02	96.00	-64.50	1.00	116	8.00	1.54184	50	0.6	100
Omega	50.011	-11.30	227.66	0.00	54.74	1.00	135	8.00	1.54184	50	0.6	100
Phi	50.011	-64.30	58.15	360.00	-57.00	1.00	360	8.00	1.54184	50	0.6	100
Phi	50.011	109.30	4.12	215.75	23.00	1.00	312	15.00	1.54184	50	0.6	100
Phi	50.011	109.30	95.73	0.00	-57.00	1.00	275	15.00	1.54184	50	0.6	100

Table 2. Crystal data and structure refinement for ted1117.

Identification code	ted1117	
Empirical formula	C ₂₉ H ₂₂ O ₅	
Formula weight	450.46	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 18.9744(16) Å	α = 90°.
	b = 8.7405(7) Å	β = 111.108(3)°.
	c = 14.7391(12) Å	γ = 90°.
Volume	2280.4(3) Å ³	
Z	4	
Density (calculated)	1.312 Mg/m ³	
Absorption coefficient	0.726 mm ⁻¹	
F(000)	944	
Crystal size	0.36 x 0.17 x 0.14 mm ³	
Theta range for data collection	2.496 to 70.208°.	
Index ranges	-22 ≤ h ≤ 22, -10 ≤ k ≤ 10, -17 ≤ l ≤ 17	
Reflections collected	27728	
Independent reflections	4310 [R(int) = 0.0438]	
Completeness to theta = 67.679°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7533 and 0.6341	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4310 / 0 / 310	
Goodness-of-fit on F ²	1.057	
Final R indices [I > 2σ(I)]	R1 = 0.0373, wR2 = 0.1007	
R indices (all data)	R1 = 0.0395, wR2 = 0.1027	
Extinction coefficient	0.0024(2)	
Largest diff. peak and hole	0.218 and -0.215 e.Å ⁻³	

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd1117. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(18)	591(1)	697(1)	7829(1)	35(1)
O(19)	1769(1)	-101(1)	8681(1)	33(1)
O(22)	3214(1)	-337(1)	8015(1)	30(1)
O(23)	3242(1)	1495(1)	9097(1)	26(1)
O(28)	4822(1)	2552(1)	6949(1)	43(1)
C(1)	984(1)	5506(2)	6641(1)	30(1)
C(2)	1344(1)	6918(2)	6732(1)	34(1)
C(3)	2100(1)	6998(2)	6832(1)	33(1)
C(4)	2508(1)	5663(1)	6845(1)	28(1)
C(5)	2157(1)	4258(1)	6765(1)	23(1)
C(6)	1392(1)	4188(1)	6664(1)	25(1)
C(7)	1082(1)	2572(1)	6556(1)	26(1)
C(8)	1207(1)	1928(1)	5668(1)	26(1)
C(9)	656(1)	1357(2)	4841(1)	32(1)
C(10)	862(1)	791(2)	4088(1)	36(1)
C(11)	1614(1)	838(2)	4167(1)	33(1)
C(12)	2168(1)	1456(1)	4992(1)	27(1)
C(13)	1966(1)	1976(1)	5749(1)	23(1)
C(14)	2496(1)	2663(1)	6724(1)	22(1)
C(15)	2333(1)	1706(1)	7515(1)	23(1)
C(16)	1603(1)	1657(1)	7417(1)	24(1)
C(17)	1261(1)	730(1)	7989(1)	25(1)
C(20)	1462(1)	-1071(2)	9242(1)	42(1)
C(21)	2966(1)	819(1)	8228(1)	22(1)
C(24)	3831(1)	638(2)	9834(1)	34(1)
C(25)	3322(1)	2619(1)	6858(1)	25(1)
C(26)	3886(1)	3237(1)	7585(1)	27(1)
C(27)	4685(1)	2980(2)	7660(1)	29(1)
C(29)	5308(1)	3214(1)	8615(1)	25(1)
C(30)	6047(1)	3332(1)	8628(1)	28(1)
C(31)	6644(1)	3481(2)	9502(1)	32(1)
C(32)	6518(1)	3494(2)	10375(1)	35(1)
C(33)	5787(1)	3374(2)	10372(1)	32(1)

C(34)

5184(1)

3253(1)

9492(1)

28(1)

Table 4. Bond lengths [Å] and angles [°] for *tc*d1117.

O(18)-C(17)	1.2073(14)	C(15)-C(21)	1.4970(15)
O(19)-C(17)	1.3372(15)	C(16)-C(17)	1.4777(16)
O(19)-C(20)	1.4464(15)	C(20)-H(20A)	0.9800
O(22)-C(21)	1.2021(14)	C(20)-H(20B)	0.9800
O(23)-C(21)	1.3345(14)	C(20)-H(20C)	0.9800
O(23)-C(24)	1.4539(14)	C(24)-H(24A)	0.9800
O(28)-C(27)	1.2263(16)	C(24)-H(24B)	0.9800
C(1)-H(1)	0.9500	C(24)-H(24C)	0.9800
C(1)-C(2)	1.3933(19)	C(25)-H(25)	0.9500
C(1)-C(6)	1.3824(17)	C(25)-C(26)	1.3269(17)
C(2)-H(2)	0.9500	C(26)-H(26)	0.9500
C(2)-C(3)	1.3909(19)	C(26)-C(27)	1.4957(16)
C(3)-H(3)	0.9500	C(27)-C(29)	1.4913(16)
C(3)-C(4)	1.3969(18)	C(29)-C(30)	1.3986(16)
C(4)-H(4)	0.9500	C(29)-C(34)	1.3955(17)
C(4)-C(5)	1.3820(17)	C(30)-H(30)	0.9500
C(5)-C(6)	1.4051(16)	C(30)-C(31)	1.3809(18)
C(5)-C(14)	1.5458(16)	C(31)-H(31)	0.9500
C(6)-C(7)	1.5166(17)	C(31)-C(32)	1.391(2)
C(7)-H(7)	1.0000	C(32)-H(32)	0.9500
C(7)-C(8)	1.5192(17)	C(32)-C(33)	1.3894(19)
C(7)-C(16)	1.5248(16)	C(33)-H(33)	0.9500
C(8)-C(9)	1.3817(17)	C(33)-C(34)	1.3908(17)
C(8)-C(13)	1.4028(16)	C(34)-H(34)	0.9500
C(9)-H(9)	0.9500		
C(9)-C(10)	1.394(2)	C(17)-O(19)-C(20)	115.19(10)
C(10)-H(10)	0.9500	C(21)-O(23)-C(24)	114.75(9)
C(10)-C(11)	1.389(2)	C(2)-C(1)-H(1)	120.5
C(11)-H(11)	0.9500	C(6)-C(1)-H(1)	120.5
C(11)-C(12)	1.3979(17)	C(6)-C(1)-C(2)	118.93(11)
C(12)-H(12)	0.9500	C(1)-C(2)-H(2)	119.8
C(12)-C(13)	1.3814(17)	C(3)-C(2)-C(1)	120.45(12)
C(13)-C(14)	1.5468(15)	C(3)-C(2)-H(2)	119.8
C(14)-C(15)	1.5546(15)	C(2)-C(3)-H(3)	119.8
C(14)-C(25)	1.5074(15)	C(2)-C(3)-C(4)	120.34(12)
C(15)-C(16)	1.3400(16)	C(4)-C(3)-H(3)	119.8

C(3)-C(4)-H(4)	120.2	C(25)-C(14)-C(15)	111.48(9)
C(5)-C(4)-C(3)	119.55(11)	C(16)-C(15)-C(14)	114.44(10)
C(5)-C(4)-H(4)	120.2	C(16)-C(15)-C(21)	126.60(10)
C(4)-C(5)-C(6)	119.69(11)	C(21)-C(15)-C(14)	118.62(9)
C(4)-C(5)-C(14)	127.56(10)	C(15)-C(16)-C(7)	114.07(10)
C(6)-C(5)-C(14)	112.68(10)	C(15)-C(16)-C(17)	127.26(11)
C(1)-C(6)-C(5)	121.04(11)	C(17)-C(16)-C(7)	118.45(10)
C(1)-C(6)-C(7)	125.48(11)	O(18)-C(17)-O(19)	123.80(11)
C(5)-C(6)-C(7)	113.45(10)	O(18)-C(17)-C(16)	123.33(11)
C(6)-C(7)-H(7)	112.9	O(19)-C(17)-C(16)	112.84(10)
C(6)-C(7)-C(8)	105.09(9)	O(19)-C(20)-H(20A)	109.5
C(6)-C(7)-C(16)	106.91(9)	O(19)-C(20)-H(20B)	109.5
C(8)-C(7)-H(7)	112.9	O(19)-C(20)-H(20C)	109.5
C(8)-C(7)-C(16)	105.49(10)	H(20A)-C(20)-H(20B)	109.5
C(16)-C(7)-H(7)	112.9	H(20A)-C(20)-H(20C)	109.5
C(9)-C(8)-C(7)	126.19(11)	H(20B)-C(20)-H(20C)	109.5
C(9)-C(8)-C(13)	120.87(11)	O(22)-C(21)-O(23)	124.58(11)
C(13)-C(8)-C(7)	112.94(10)	O(22)-C(21)-C(15)	122.84(10)
C(8)-C(9)-H(9)	120.3	O(23)-C(21)-C(15)	112.50(9)
C(8)-C(9)-C(10)	119.40(12)	O(23)-C(24)-H(24A)	109.5
C(10)-C(9)-H(9)	120.3	O(23)-C(24)-H(24B)	109.5
C(9)-C(10)-H(10)	120.1	O(23)-C(24)-H(24C)	109.5
C(11)-C(10)-C(9)	119.79(11)	H(24A)-C(24)-H(24B)	109.5
C(11)-C(10)-H(10)	120.1	H(24A)-C(24)-H(24C)	109.5
C(10)-C(11)-H(11)	119.6	H(24B)-C(24)-H(24C)	109.5
C(10)-C(11)-C(12)	120.84(12)	C(14)-C(25)-H(25)	116.8
C(12)-C(11)-H(11)	119.6	C(26)-C(25)-C(14)	126.47(11)
C(11)-C(12)-H(12)	120.4	C(26)-C(25)-H(25)	116.8
C(13)-C(12)-C(11)	119.27(12)	C(25)-C(26)-H(26)	120.0
C(13)-C(12)-H(12)	120.4	C(25)-C(26)-C(27)	120.01(11)
C(8)-C(13)-C(14)	113.23(10)	C(27)-C(26)-H(26)	120.0
C(12)-C(13)-C(8)	119.77(10)	O(28)-C(27)-C(26)	120.25(11)
C(12)-C(13)-C(14)	126.99(10)	O(28)-C(27)-C(29)	120.56(11)
C(5)-C(14)-C(13)	104.26(9)	C(29)-C(27)-C(26)	119.17(10)
C(5)-C(14)-C(15)	104.89(9)	C(30)-C(29)-C(27)	118.27(11)
C(13)-C(14)-C(15)	104.46(9)	C(34)-C(29)-C(27)	122.40(10)
C(25)-C(14)-C(5)	116.46(9)	C(34)-C(29)-C(30)	119.27(11)
C(25)-C(14)-C(13)	114.15(9)	C(29)-C(30)-H(30)	120.0

C(31)-C(30)-C(29)	120.05(12)	C(33)-C(32)-H(32)	120.0
C(31)-C(30)-H(30)	120.0	C(32)-C(33)-H(33)	120.2
C(30)-C(31)-H(31)	119.8	C(32)-C(33)-C(34)	119.57(12)
C(30)-C(31)-C(32)	120.46(12)	C(34)-C(33)-H(33)	120.2
C(32)-C(31)-H(31)	119.8	C(29)-C(34)-H(34)	119.7
C(31)-C(32)-H(32)	120.0	C(33)-C(34)-C(29)	120.57(11)
C(33)-C(32)-C(31)	120.06(12)	C(33)-C(34)-H(34)	119.7

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tcd1117. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(18)	22(1)	52(1)	33(1)	7(1)	11(1)	-6(1)
O(19)	28(1)	41(1)	30(1)	12(1)	12(1)	0(1)
O(22)	30(1)	30(1)	30(1)	-5(1)	11(1)	3(1)
O(23)	25(1)	30(1)	21(1)	-2(1)	4(1)	2(1)
O(28)	25(1)	78(1)	28(1)	-6(1)	11(1)	1(1)
C(1)	26(1)	42(1)	23(1)	3(1)	9(1)	7(1)
C(2)	37(1)	36(1)	26(1)	-2(1)	8(1)	10(1)
C(3)	38(1)	30(1)	27(1)	-5(1)	7(1)	-2(1)
C(4)	25(1)	32(1)	24(1)	-3(1)	6(1)	-3(1)
C(5)	22(1)	30(1)	18(1)	-1(1)	6(1)	0(1)
C(6)	21(1)	34(1)	19(1)	3(1)	7(1)	1(1)
C(7)	17(1)	36(1)	25(1)	5(1)	7(1)	-2(1)
C(8)	23(1)	28(1)	24(1)	5(1)	6(1)	-4(1)
C(9)	26(1)	35(1)	28(1)	6(1)	1(1)	-8(1)
C(10)	40(1)	34(1)	23(1)	0(1)	0(1)	-10(1)
C(11)	45(1)	32(1)	22(1)	0(1)	10(1)	-4(1)
C(12)	30(1)	28(1)	23(1)	2(1)	9(1)	-3(1)
C(13)	23(1)	24(1)	21(1)	2(1)	6(1)	-3(1)
C(14)	20(1)	27(1)	20(1)	0(1)	7(1)	-2(1)
C(15)	21(1)	27(1)	20(1)	-1(1)	7(1)	-2(1)
C(16)	20(1)	32(1)	22(1)	1(1)	7(1)	-2(1)
C(17)	24(1)	31(1)	21(1)	-2(1)	8(1)	-4(1)
C(20)	44(1)	48(1)	36(1)	14(1)	18(1)	-3(1)
C(21)	20(1)	27(1)	21(1)	-2(1)	9(1)	-4(1)
C(24)	33(1)	37(1)	25(1)	3(1)	1(1)	2(1)
C(25)	21(1)	31(1)	24(1)	1(1)	10(1)	-1(1)
C(26)	20(1)	35(1)	27(1)	-1(1)	9(1)	0(1)
C(27)	22(1)	38(1)	27(1)	2(1)	10(1)	-2(1)
C(29)	20(1)	26(1)	28(1)	2(1)	8(1)	0(1)
C(30)	22(1)	30(1)	32(1)	2(1)	11(1)	-2(1)
C(31)	21(1)	35(1)	40(1)	-2(1)	9(1)	-2(1)
C(32)	28(1)	37(1)	33(1)	-4(1)	1(1)	-1(1)
C(33)	34(1)	34(1)	29(1)	-1(1)	11(1)	1(1)

C(34)	23(1)	30(1)	30(1)	0(1)	11(1)	-1(1)
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Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for tcd1117.

	x	y	z	U(eq)
H(1)	465	5451	6565	36
H(2)	1071	7832	6725	41
H(3)	2341	7966	6893	40
H(4)	3024	5720	6907	33
H(7)	541	2532	6499	31
H(9)	142	1350	4787	38
H(10)	490	375	3523	43
H(11)	1753	445	3654	40
H(12)	2678	1518	5030	32
H(20A)	1100	-1791	8807	62
H(20B)	1204	-438	9576	62
H(20C)	1872	-1641	9724	62
H(24A)	4212	308	9570	51
H(24B)	3608	-262	10024	51
H(24C)	4068	1289	10404	51
H(25)	3451	2097	6374	30
H(26)	3783	3844	8058	33
H(30)	6138	3308	8035	33
H(31)	7144	3575	9507	39
H(32)	6931	3585	10973	42
H(33)	5699	3375	10967	39
H(34)	4682	3197	9487	33

Table 7. Torsion angles [°] for tcd1117.

O(28)-C(27)-C(29)-C(30)	16.55(18)	C(8)-C(13)-C(14)-C(5)	-56.12(12)
O(28)-C(27)-C(29)-C(34)	-160.61(13)	C(8)-C(13)-C(14)-C(15)	53.72(12)
C(1)-C(2)-C(3)-C(4)	-0.07(19)	C(8)-C(13)-C(14)-C(25)	175.73(10)
C(1)-C(6)-C(7)-C(8)	120.36(12)	C(9)-C(8)-C(13)-C(12)	0.46(17)
C(1)-C(6)-C(7)-C(16)	-127.86(12)	C(9)-C(8)-C(13)-C(14)	179.58(11)
C(2)-C(1)-C(6)-C(5)	-0.69(17)	C(9)-C(10)-C(11)-C(12)	-0.42(19)
C(2)-C(1)-C(6)-C(7)	-178.77(11)	C(10)-C(11)-C(12)-C(13)	2.29(18)
C(2)-C(3)-C(4)-C(5)	-0.60(18)	C(11)-C(12)-C(13)-C(8)	-2.29(17)
C(3)-C(4)-C(5)-C(6)	0.61(17)	C(11)-C(12)-C(13)-C(14)	178.72(11)
C(3)-C(4)-C(5)-C(14)	177.44(11)	C(12)-C(13)-C(14)-C(5)	122.93(12)
C(4)-C(5)-C(6)-C(1)	0.03(17)	C(12)-C(13)-C(14)-C(15)	-127.23(12)
C(4)-C(5)-C(6)-C(7)	178.33(10)	C(12)-C(13)-C(14)-C(25)	-5.22(16)
C(4)-C(5)-C(14)-C(13)	-121.88(12)	C(13)-C(8)-C(9)-C(10)	1.42(18)
C(4)-C(5)-C(14)-C(15)	128.60(12)	C(13)-C(14)-C(15)-C(16)	-54.14(13)
C(4)-C(5)-C(14)-C(25)	4.86(17)	C(13)-C(14)-C(15)-C(21)	119.60(10)
C(5)-C(6)-C(7)-C(8)	-57.85(12)	C(13)-C(14)-C(25)-C(26)	174.21(12)
C(5)-C(6)-C(7)-C(16)	53.92(12)	C(14)-C(5)-C(6)-C(1)	-177.24(10)
C(5)-C(14)-C(15)-C(16)	55.24(12)	C(14)-C(5)-C(6)-C(7)	1.06(13)
C(5)-C(14)-C(15)-C(21)	-131.02(10)	C(14)-C(15)-C(16)-C(7)	-0.92(15)
C(5)-C(14)-C(25)-C(26)	52.55(16)	C(14)-C(15)-C(16)-C(17)	173.52(11)
C(6)-C(1)-C(2)-C(3)	0.70(18)	C(14)-C(15)-C(21)-O(22)	-73.79(14)
C(6)-C(5)-C(14)-C(13)	55.13(12)	C(14)-C(15)-C(21)-O(23)	103.11(11)
C(6)-C(5)-C(14)-C(15)	-54.38(11)	C(14)-C(25)-C(26)-C(27)	174.77(11)
C(6)-C(5)-C(14)-C(25)	-178.13(9)	C(15)-C(14)-C(25)-C(26)	-67.73(15)
C(6)-C(7)-C(8)-C(9)	-122.33(12)	C(15)-C(16)-C(17)-O(18)	-176.68(12)
C(6)-C(7)-C(8)-C(13)	56.88(12)	C(15)-C(16)-C(17)-O(19)	1.41(18)
C(6)-C(7)-C(16)-C(15)	-54.54(13)	C(16)-C(7)-C(8)-C(9)	124.88(12)
C(6)-C(7)-C(16)-C(17)	130.50(11)	C(16)-C(7)-C(8)-C(13)	-55.91(12)
C(7)-C(8)-C(9)-C(10)	-179.43(12)	C(16)-C(15)-C(21)-O(22)	99.11(15)
C(7)-C(8)-C(13)-C(12)	-178.80(10)	C(16)-C(15)-C(21)-O(23)	-83.99(14)
C(7)-C(8)-C(13)-C(14)	0.33(14)	C(20)-O(19)-C(17)-O(18)	0.06(18)
C(7)-C(16)-C(17)-O(18)	-2.46(18)	C(20)-O(19)-C(17)-C(16)	-178.01(11)
C(7)-C(16)-C(17)-O(19)	175.63(10)	C(21)-C(15)-C(16)-C(7)	-174.07(10)
C(8)-C(7)-C(16)-C(15)	56.96(13)	C(21)-C(15)-C(16)-C(17)	0.4(2)
C(8)-C(7)-C(16)-C(17)	-118.00(11)	C(24)-O(23)-C(21)-O(22)	-6.46(16)
C(8)-C(9)-C(10)-C(11)	-1.43(19)	C(24)-O(23)-C(21)-C(15)	176.71(10)

C(25)-C(14)-C(15)-C(16)	-177.89(10)	C(27)-C(29)-C(34)-C(33)	175.77(12)
C(25)-C(14)-C(15)-C(21)	-4.14(14)	C(29)-C(30)-C(31)-C(32)	0.91(19)
C(25)-C(26)-C(27)-O(28)	18.72(19)	C(30)-C(29)-C(34)-C(33)	-1.36(18)
C(25)-C(26)-C(27)-C(29)	-159.81(12)	C(30)-C(31)-C(32)-C(33)	-0.7(2)
C(26)-C(27)-C(29)-C(30)	-164.92(11)	C(31)-C(32)-C(33)-C(34)	-0.5(2)
C(26)-C(27)-C(29)-C(34)	17.92(18)	C(32)-C(33)-C(34)-C(29)	1.55(19)
C(27)-C(29)-C(30)-C(31)	-177.12(12)	C(34)-C(29)-C(30)-C(31)	0.13(18)

Table 8. Hydrogen bonds for tcd1117 [$\text{\AA}$ and $^\circ$].

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
C(3)-H(3)...O(22)#1	0.95	2.39	3.2060(15)	144

Symmetry transformations used to generate equivalent atoms:

#1 x,y+1,z