Supporting Information

A Safer and Convenient Synthesis of 3,4-Dicyanofuroxan

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Supporting Information Available:

A. Crystal Data and Structure Refinement

Table S1: Crystal data and structure refinement for compound **1**.

Empirical formula	$C_4N_4O_2$
Formula weight	136.07
Temperature/K	250.00(10)
Crystal system	orthorhombic
Space group	$Pna2_1$
a/Å	10.1798(5)
b/Å	10.8181(4)
c/Å	10.1946(4)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1122.69(8)
Z	8
ρ_{calc}g/cm^3	1.6099
μ/mm^{-1}	0.135
F(000)	544.3
Crystal size/mm^3	0.34 × 0.32 × 0.18
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	5.5 to 52.72
Index ranges	$-8 \leq h \leq 12$, $-13 \leq k \leq 13$, $-12 \leq l \leq 12$
Reflections collected	4937
Independent reflections	2179 [R_{int} = 0.0201, R_{sigma} = 0.0242]
Data/restraints/parameters	2179/0/180
Goodness-of-fit on F^2	1.060
Final R indexes [I>=2σ (I)]	R_1 = 0.0382, wR_2 = 0.0895
Final R indexes [all data]	R_1 = 0.0425, wR_2 = 0.0922
Largest diff. peak/hole / e Å^{-3}	0.22/-0.16
Flack parameter	0(2)

B. Geometric Parameters

Table S2: Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for compound **1**. U_{eq} is defined as 1/3 of the trace of the orthogonalized U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	3492(2)	6178.7(18)	5672(2)	35.3(5)
C2	4664(2)	6523.2(18)	6298(2)	36.9(5)
C3	5168(2)	6023(2)	7495(2)	39.0(5)
C4	2539(2)	5275(2)	6004(2)	42.8(6)
N1	3398.9(19)	6850.8(16)	4576.6(19)	38.8(4)
N2	5297(2)	7372.5(17)	5653(2)	44.6(5)
N3	5575(2)	5592(2)	8426(2)	56.9(6)
N4	1794(2)	4537(2)	6272(3)	62.2(6)
O1	4574.0(17)	7621.5(15)	4554.7(17)	49.2(4)
O2	2630.7(17)	6938.1(18)	3683.2(17)	55.0(5)
C1A	8611(2)	3598.3(19)	5812(2)	39.4(5)
C2A	8527(2)	4745(2)	6434(2)	42.1(6)
C3A	9179(3)	5125(2)	7600(3)	46.7(6)
C4A	9391(3)	2536(2)	6100(2)	47.0(6)
N1A	7843(2)	3650.4(18)	4739(2)	45.3(5)
N2A	7756(2)	5497.6(17)	5811(2)	48.1(5)
N3A	9697(3)	5461(3)	8509(3)	74.2(7)
N4A	10028(3)	1702.5(19)	6321(2)	62.2(6)
O1A	7297.0(17)	4891.1(17)	4742(2)	55.9(5)
O2A	7552.6(18)	2973.0(19)	3845.8(19)	65.0(6)

Table S3: Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for compound **1**. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{12}**	**U_{13}**	**U_{23}**
C1	37.3(12)	35.3(10)	33.3(10)	3.6(9)	3.7(10)	-4.5(8)
C2	34.9(13)	35.6(10)	40.0(11)	4.8(9)	3.9(10)	-5(1)
C3	38.3(13)	41.1(11)	37.7(12)	0.9(10)	0.7(11)	-7(1)
C4	40.5(15)	45.1(11)	42.7(13)	-0.2(10)	0.3(11)	-2.4(10)
N1	39.2(11)	41.1(9)	36.2(9)	2.7(8)	4.3(9)	-2.9(8)
N2	42.2(11)	43.3(10)	48.4(11)	-4.6(8)	-0.9(10)	1.2(9)
N3	66.5(16)	59.8(13)	44.5(11)	6.5(12)	-6.1(11)	-2.1(10)
N4	53.1(15)	64.7(14)	68.7(14)	-15.0(12)	6.0(13)	5.8(13)
O1	48.7(10)	49.2(9)	49.6(9)	-3.6(8)	3.2(9)	9.6(8)
O2	54.8(11)	69.9(12)	40.2(9)	5.0(9)	-11.1(9)	4.8(9)
C1A	38.1(13)	42.7(11)	37.3(11)	-2.7(10)	7.4(10)	2.9(9)
C2A	36.4(14)	47.5(12)	42.3(12)	-0.7(10)	7.6(11)	4.7(10)
C3A	41.7(14)	53.7(13)	44.5(13)	-4.1(12)	7.4(12)	-1.4(11)
C4A	56.2(16)	40.2(11)	44.5(13)	1.3(11)	-0.3(13)	1.6(10)
N1A	40.5(12)	50.3(11)	45.2(11)	2.6(9)	1.8(10)	1.4(10)
N2A	41.7(12)	48.1(11)	54.5(13)	10.2(9)	1.2(10)	-0.5(9)
N3A	65.8(16)	99.3(19)	57.6(15)	-17.5(16)	4.8(14)	-12.7(14)
N4A	79.0(17)	46.4(11)	61.3(14)	11.2(12)	-12.3(14)	0.4(12)
O1A	48.7(11)	61.6(10)	57.4(10)	9.5(9)	-9.3(9)	6.4(10)
O2A	66.4(13)	71.6(13)	56.9(12)	1.4(10)	-14.4(10)	-15.3(11)

Table S4: Bond Lengths (Å) for compound **1**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.404(3)	C1A	C2A	1.396(3)
C1	C4	1.418(3)	C1A	C4A	1.427(3)
C1	N1	1.336(3)	C1A	N1A	1.346(3)
C2	C3	1.430(3)	C2A	C3A	1.422(4)
C2	N2	1.301(3)	C2A	N2A	1.296(3)
C3	N3	1.136(3)	C3A	N3A	1.127(4)
C4	N4	1.135(3)	C4A	N4A	1.133(3)
N1	O1	1.458(2)	N1A	O1A	1.453(3)
N1	O2	1.204(2)	N1A	O2A	1.205(3)
N2	O1	1.366(3)	N2A	O1A	1.356(3)

Table S5: Bond Angles (degree) for compound **1**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C4	C1	C2	131.0(2)	C4A	C1A	C2A	131.0(2)
N1	C1	C2	107.24(19)	N1A	C1A	C2A	107.2(2)
N1	C1	C4	121.8(2)	N1A	C1A	C4A	121.6(2)
C3	C2	C1	126.3(2)	C3A	C2A	C1A	127.4(2)
N2	C2	C1	112.2(2)	N2A	C2A	C1A	111.9(2)
N2	C2	C3	121.4(2)	N2A	C2A	C3A	120.7(2)
N3	C3	C2	177.9(2)	N3A	C3A	C2A	177.9(3)
N4	C4	C1	178.8(3)	N4A	C4A	C1A	178.8(3)
O1	N1	C1	105.40(18)	O1A	N1A	C1A	105.00(18)
O2	N1	C1	136.2(2)	O2A	N1A	C1A	137.0(2)
O2	N1	O1	118.44(18)	O2A	N1A	O1A	118.0(2)
O1	N2	C2	106.69(18)	O1A	N2A	C2A	107.36(17)
N2	O1	N1	108.45(15)	N2A	O1A	N1A	108.49(16)

C. Special Details

Geometry. All experimental estimated standard deviations (esds) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances and angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.