Supplementary Data for:

Synthesis of Diazeniumdiolates from the Reactions of Nitric Oxide with Enolates

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Table S1. Crystal data and structure refinement for **14b**.

Identification code	na105	
Empirical formula	C ₈ H ₁₂ K ₂ N ₄ O ₅	
Formula weight	322.42	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 13.5041(17) Å	α = 90°.
	b = 6.7097(7) Å	β = 109.973(14)°.
	c = 14.271(4) Å	γ = 90°.
Volume	1215.3(4) Å ³	
Z	4	
Density (calculated)	1.762 Mg/m ³	
Absorption coefficient	0.803 mm ⁻¹	
F(000)	664	
Crystal size	0.60 x 0.40 x 0.10 mm ³	
Theta range for data collection	2.91 to 25.00°.	
Index ranges	-1 ≤ h ≤ 16, -7 ≤ k ≤ 1, -16 ≤ l ≤ 16	
Reflections collected	2827	
Independent reflections	2107 [R(int) = 0.0376]	
Completeness to theta = 25.00°	99.1 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2107 / 0 / 220	
Goodness-of-fit on F ²	1.078	
Final R indices [I > 2σ(I)]	R1 = 0.0335, wR2 = 0.0896	
R indices (all data)	R1 = 0.0404, wR2 = 0.0958	
Largest diff. peak and hole	0.431 and -0.499 e.Å ⁻³	

Table S2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **14b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
K(1)	10348(1)	-2494(1)	112(1)	16(1)
K(2)	10473(1)	-2483(1)	2653(1)	14(1)
O(1)	8908(1)	-740(2)	1014(1)	17(1)
N(1)	8065(1)	-1864(2)	779(1)	12(1)
N(2)	8055(1)	-3783(2)	678(1)	14(1)
O(2)	8983(1)	-4545(2)	853(1)	17(1)
O(3)	8885(1)	-721(2)	3267(1)	18(1)
N(3)	8042(1)	-1837(2)	2952(1)	12(1)
N(4)	8024(1)	-3752(2)	3049(1)	15(1)
O(4)	8950(1)	-4522(2)	3487(1)	18(1)
O(5)	7231(1)	2111(2)	1578(1)	26(1)
C(1)	7128(1)	328(2)	1554(1)	13(1)
C(2)	7039(1)	-839(2)	590(1)	12(1)
C(3)	6103(1)	-2292(3)	338(1)	16(1)
C(4)	6043(1)	-3533(3)	1211(1)	18(1)
C(5)	6075(1)	-2221(3)	2091(1)	17(1)
C(6)	7020(1)	-797(2)	2458(1)	13(1)
C(7)	6906(1)	637(3)	-256(1)	19(1)
C(8)	6877(1)	705(3)	3206(1)	20(1)

Table S3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for **14b**.

K(1)-O(2)#1	2.7395(12)
K(1)-O(4)#2	2.7538(13)
K(1)-O(2)	2.7813(13)
K(1)-O(4)#3	2.7895(14)
K(1)-O(1)	2.9208(12)
K(1)-O(3)#3	2.9518(13)
K(1)-O(1)#4	3.0667(13)
K(1)-O(3)#5	3.0787(13)
K(1)-K(1)#4	3.4619(7)
K(1)-K(1)#1	3.4761(7)
K(1)-K(2)#3	3.5706(10)
K(1)-K(2)	3.5720(10)
K(2)-O(2)#2	2.8113(13)
K(2)-O(1)	2.8148(13)
K(2)-O(3)#5	2.8237(13)
K(2)-O(1)#5	2.8300(13)
K(2)-O(3)	2.8366(13)
K(2)-O(4)#2	2.8394(13)
K(2)-O(5)#5	2.9266(16)
K(2)-O(2)	3.0049(14)
K(2)-O(4)	3.0311(13)
K(2)-N(1)	3.4578(15)
K(2)-K(2)#5	3.5632(4)
O(1)-N(1)	1.3106(18)
N(1)-N(2)	1.295(2)
N(1)-C(2)	1.487(2)
N(2)-O(2)	1.2971(18)
O(3)-N(3)	1.3077(18)
N(3)-N(4)	1.293(2)
N(3)-C(6)	1.491(2)
N(4)-O(4)	1.2991(18)
O(5)-C(1)	1.203(2)
C(1)-C(6)	1.544(2)
C(1)-C(2)	1.552(2)

C(2)-C(7)	1.523(2)
C(2)-C(3)	1.538(2)
C(3)-C(4)	1.524(2)
C(3)-H(3A)	1.012(19)
C(3)-H(3B)	0.99(2)
C(4)-C(5)	1.522(2)
C(4)-H(4A)	1.01(2)
C(4)-H(4B)	1.01(2)
C(5)-C(6)	1.535(2)
C(5)-H(5A)	1.01(2)
C(5)-H(5B)	0.95(2)
C(6)-C(8)	1.526(2)
C(7)-H(7A)	0.93(2)
C(7)-H(7B)	0.97(2)
C(7)-H(7C)	1.00(2)
C(8)-H(8A)	0.95(2)
C(8)-H(8B)	0.99(2)
C(8)-H(8C)	1.00(2)

O(2)#1-K(1)-O(4)#2	143.07(4)
O(2)#1-K(1)-O(2)	101.96(3)
O(4)#2-K(1)-O(2)	100.98(4)
O(2)#1-K(1)-O(4)#3	100.34(4)
O(4)#2-K(1)-O(4)#3	102.71(3)
O(2)-K(1)-O(4)#3	102.02(4)
O(2)#1-K(1)-O(1)	153.76(4)
O(4)#2-K(1)-O(1)	60.24(4)
O(2)-K(1)-O(1)	53.48(4)
O(4)#3-K(1)-O(1)	78.94(4)
O(2)#1-K(1)-O(3)#3	59.53(4)
O(4)#2-K(1)-O(3)#3	154.30(4)
O(2)-K(1)-O(3)#3	79.06(4)
O(4)#3-K(1)-O(3)#3	53.02(4)
O(1)-K(1)-O(3)#3	102.32(4)
O(2)#1-K(1)-O(1)#4	91.53(4)
O(4)#2-K(1)-O(1)#4	76.99(4)

O(2)-K(1)-O(1)#4	158.03(3)
O(4)#3-K(1)-O(1)#4	58.04(3)
O(1)-K(1)-O(1)#4	109.40(3)
O(3)#3-K(1)-O(1)#4	93.37(4)
O(2)#1-K(1)-O(3)#5	77.50(4)
O(4)#2-K(1)-O(3)#5	91.11(4)
O(2)-K(1)-O(3)#5	57.51(3)
O(4)#3-K(1)-O(3)#5	157.59(3)
O(1)-K(1)-O(3)#5	93.20(4)
O(3)#3-K(1)-O(3)#5	109.64(3)
O(1)#4-K(1)-O(3)#5	143.63(3)
O(2)#2-K(2)-O(1)	105.45(4)
O(2)#2-K(2)-O(3)#5	148.22(4)
O(1)-K(2)-O(3)#5	101.31(4)
O(2)#2-K(2)-O(1)#5	95.21(4)
O(1)-K(2)-O(1)#5	147.64(4)
O(3)#5-K(2)-O(1)#5	69.58(4)
O(2)#2-K(2)-O(3)	60.17(4)
O(1)-K(2)-O(3)	69.61(4)
O(3)#5-K(2)-O(3)	148.07(4)
O(1)#5-K(2)-O(3)	101.23(4)
O(2)#2-K(2)-O(4)#2	83.81(4)
O(1)-K(2)-O(4)#2	60.57(4)
O(3)#5-K(2)-O(4)#2	94.85(4)
O(1)#5-K(2)-O(4)#2	148.17(4)
O(3)-K(2)-O(4)#2	105.64(4)
O(2)#2-K(2)-O(5)#5	79.17(4)
O(1)-K(2)-O(5)#5	138.47(4)
O(3)#5-K(2)-O(5)#5	69.42(4)
O(1)#5-K(2)-O(5)#5	69.16(3)
O(3)-K(2)-O(5)#5	137.61(4)
O(4)#2-K(2)-O(5)#5	79.48(4)
O(2)#2-K(2)-O(2)	153.70(3)
O(1)-K(2)-O(2)	52.25(4)
O(3)#5-K(2)-O(2)	58.02(3)
O(1)#5-K(2)-O(2)	100.17(4)

O(3)-K(2)-O(2)	95.74(4)
O(4)#2-K(2)-O(2)	93.85(4)
O(5)#5-K(2)-O(2)	126.28(4)
O(2)#2-K(2)-O(4)	93.15(3)
O(1)-K(2)-O(4)	95.52(4)
O(3)#5-K(2)-O(4)	101.08(4)
O(1)#5-K(2)-O(4)	58.10(3)
O(3)-K(2)-O(4)	51.75(4)
O(4)#2-K(2)-O(4)	153.66(3)
O(5)#5-K(2)-O(4)	125.76(4)
O(2)-K(2)-O(4)	77.41(4)
O(2)#2-K(2)-N(1)	115.81(4)
O(1)-K(2)-N(1)	21.09(3)
O(3)#5-K(2)-N(1)	95.16(4)
O(1)#5-K(2)-N(1)	126.60(4)
O(3)-K(2)-N(1)	65.07(4)
O(4)#2-K(2)-N(1)	80.95(4)
O(5)#5-K(2)-N(1)	153.79(4)
O(2)-K(2)-N(1)	38.39(3)
O(4)-K(2)-N(1)	76.87(3)
O(2)#2-K(2)-K(2)#5	137.96(3)
O(1)-K(2)-K(2)#5	98.83(3)
O(3)#5-K(2)-K(2)#5	51.15(3)
O(1)#5-K(2)-K(2)#5	50.67(3)
O(3)-K(2)-K(2)#5	98.79(3)
O(4)#2-K(2)-K(2)#5	138.22(3)
O(5)#5-K(2)-K(2)#5	104.34(3)
O(2)-K(2)-K(2)#5	49.80(3)
O(4)-K(2)-K(2)#5	50.22(3)
C(1)#5-K(2)-K(2)#5	83.94(3)
N(1)-K(2)-K(2)#5	79.36(3)
N(1)-O(1)-K(2)	108.29(9)
N(1)-O(1)-K(2)#2	130.37(9)
K(2)-O(1)-K(2)#2	78.28(3)
N(1)-O(1)-K(1)	107.83(9)
K(2)-O(1)-K(1)	77.02(3)

K(2)#2-O(1)-K(1)	121.31(4)
N(1)-O(1)-K(1)#4	133.20(9)
K(2)-O(1)-K(1)#4	116.23(4)
K(2)#2-O(1)-K(1)#4	74.41(3)
K(1)-O(1)-K(1)#4	70.60(3)
N(2)-N(1)-O(1)	125.13(13)
N(2)-N(1)-C(2)	117.79(13)
O(1)-N(1)-C(2)	117.06(13)
N(2)-N(1)-K(2)	86.44(9)
O(1)-N(1)-K(2)	50.61(7)
C(2)-N(1)-K(2)	140.16(9)
N(1)-N(2)-O(2)	113.47(13)
N(2)-O(2)-K(1)#1	130.72(9)
N(2)-O(2)-K(1)	117.00(9)
K(1)#1-O(2)-K(1)	78.04(3)
N(2)-O(2)-K(2)#5	114.03(9)
K(1)#1-O(2)-K(2)#5	80.06(4)
K(1)-O(2)-K(2)#5	126.88(4)
N(2)-O(2)-K(2)	107.67(9)
K(1)#1-O(2)-K(2)	121.61(4)
K(1)-O(2)-K(2)	76.13(4)
K(2)#5-O(2)-K(2)	75.48(3)
N(3)-O(3)-K(2)#2	130.56(9)
N(3)-O(3)-K(2)	108.47(9)
K(2)#2-O(3)-K(2)	78.03(3)
N(3)-O(3)-K(1)#6	108.27(9)
K(2)#2-O(3)-K(1)#6	120.56(4)
K(2)-O(3)-K(1)#6	76.14(3)
N(3)-O(3)-K(1)#2	133.72(9)
K(2)#2-O(3)-K(1)#2	74.34(3)
K(2)-O(3)-K(1)#2	115.43(4)
K(1)#6-O(3)-K(1)#2	70.36(3)
N(4)-N(3)-O(3)	125.27(13)
N(4)-N(3)-C(6)	117.89(13)
O(3)-N(3)-C(6)	116.83(13)
N(3)-N(4)-O(4)	113.37(13)

N(4)-O(4)-K(1)#5	132.31(10)
N(4)-O(4)-K(1)#6	118.25(9)
K(1)#5-O(4)-K(1)#6	77.29(3)
N(4)-O(4)-K(2)#5	114.25(9)
K(1)#5-O(4)-K(2)#5	79.36(4)
K(1)#6-O(4)-K(2)#5	125.13(4)
N(4)-O(4)-K(2)	107.63(9)
K(1)#5-O(4)-K(2)	120.04(4)
K(1)#6-O(4)-K(2)	75.55(4)
K(2)#5-O(4)-K(2)	74.66(3)
C(1)-O(5)-K(2)#2	101.59(11)
O(5)-C(1)-C(6)	120.36(15)
O(5)-C(1)-C(2)	119.98(15)
C(6)-C(1)-C(2)	119.57(13)
O(5)-C(1)-K(2)#2	58.01(10)
C(6)-C(1)-K(2)#2	106.64(9)
C(2)-C(1)-K(2)#2	106.96(9)
N(1)-C(2)-C(7)	107.11(12)
N(1)-C(2)-C(3)	112.92(13)
C(7)-C(2)-C(3)	111.21(13)
N(1)-C(2)-C(1)	106.67(12)
C(7)-C(2)-C(1)	109.11(13)
C(3)-C(2)-C(1)	109.65(13)
C(4)-C(3)-C(2)	115.07(14)
C(4)-C(3)-H(3A)	109.0(10)
C(2)-C(3)-H(3A)	104.7(10)
C(4)-C(3)-H(3B)	111.4(13)
C(2)-C(3)-H(3B)	109.2(12)
H(3A)-C(3)-H(3B)	106.9(16)
C(5)-C(4)-C(3)	111.36(14)
C(5)-C(4)-H(4A)	110.8(11)
C(3)-C(4)-H(4A)	110.5(11)
C(5)-C(4)-H(4B)	109.6(11)
C(3)-C(4)-H(4B)	107.4(11)
H(4A)-C(4)-H(4B)	107.0(15)
C(4)-C(5)-C(6)	115.32(14)

C(4)-C(5)-H(5A)	109.4(11)
C(6)-C(5)-H(5A)	105.5(11)
C(4)-C(5)-H(5B)	109.4(14)
C(6)-C(5)-H(5B)	109.9(14)
H(5A)-C(5)-H(5B)	107.0(17)
N(3)-C(6)-C(8)	107.23(13)
N(3)-C(6)-C(5)	113.37(13)
C(8)-C(6)-C(5)	111.16(14)
N(3)-C(6)-C(1)	106.65(12)
C(8)-C(6)-C(1)	109.36(14)
C(5)-C(6)-C(1)	108.91(13)
C(2)-C(7)-H(7A)	108.9(13)
C(2)-C(7)-H(7B)	109.8(13)
H(7A)-C(7)-H(7B)	111.4(18)
C(2)-C(7)-H(7C)	111.0(13)
H(7A)-C(7)-H(7C)	106.5(17)
H(7B)-C(7)-H(7C)	109.3(17)
C(6)-C(8)-H(8A)	108.5(13)
C(6)-C(8)-H(8B)	111.8(14)
H(8A)-C(8)-H(8B)	106.9(18)
C(6)-C(8)-H(8C)	110.3(12)
H(8A)-C(8)-H(8C)	111.5(18)
H(8B)-C(8)-H(8C)	107.8(19)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y-1,-z #2 -x+2,y+1/2,-z+1/2 #3 x,-y-1/2,z-1/2

#4 -x+2,-y,-z #5 -x+2,y-1/2,-z+1/2 #6 x,-y-1/2,z+1/2

Table S4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **14b**. 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}
K(1)	19(1)	13(1)	17(1)	-3(1)	6(1)	0(1)
K(2)	16(1)	11(1)	16(1)	0(1)	5(1)	2(1)
O(1)	11(1)	14(1)	23(1)	-3(1)	4(1)	-2(1)
N(1)	14(1)	10(1)	12(1)	-2(1)	4(1)	0(1)
N(2)	16(1)	11(1)	16(1)	0(1)	6(1)	4(1)
O(2)	15(1)	15(1)	22(1)	1(1)	8(1)	6(1)
O(3)	11(1)	16(1)	23(1)	2(1)	2(1)	-3(1)
N(3)	14(1)	10(1)	13(1)	2(1)	4(1)	0(1)
N(4)	17(1)	12(1)	15(1)	3(1)	4(1)	4(1)
O(4)	17(1)	16(1)	18(1)	4(1)	2(1)	7(1)
O(5)	44(1)	9(1)	27(1)	0(1)	14(1)	2(1)
C(1)	11(1)	10(1)	16(1)	1(1)	4(1)	2(1)
C(2)	12(1)	11(1)	13(1)	1(1)	4(1)	3(1)
C(3)	12(1)	16(1)	17(1)	-3(1)	2(1)	1(1)
C(4)	13(1)	12(1)	26(1)	-1(1)	4(1)	-2(1)
C(5)	14(1)	17(1)	20(1)	5(1)	6(1)	0(1)
C(6)	12(1)	12(1)	14(1)	1(1)	4(1)	4(1)
C(7)	20(1)	21(1)	16(1)	6(1)	5(1)	7(1)
C(8)	21(1)	21(1)	18(1)	-2(1)	8(1)	6(1)

Table S5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **14b**.

	x	y	z	U(eq)
H(3A)	5456(14)	-1410(30)	89(13)	14(4)
H(3B)	6103(16)	-3140(30)	-227(16)	26(5)
H(4A)	6626(15)	-4550(30)	1414(14)	18(5)
H(4B)	5357(17)	-4290(30)	972(15)	26(5)
H(5A)	5433(15)	-1340(30)	1896(14)	22(5)
H(5B)	6047(17)	-3030(30)	2625(17)	30(6)
H(7A)	6254(17)	1250(30)	-413(15)	23(5)
H(7B)	7474(17)	1610(30)	-58(16)	29(5)
H(7C)	6909(16)	-60(30)	-874(16)	30(6)
H(8A)	6205(17)	1300(30)	2928(15)	28(5)
H(8B)	6898(18)	50(40)	3836(18)	41(6)
H(8C)	7448(17)	1720(30)	3372(16)	30(5)