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Title : An Unusual Reaction of Chloramine-T with Araldoximes

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Figure S1

KV-1

Figure S2

KV-1

21.786
21.276

77.428
77.000
76.581

96.222

144.551
139.312
138.094
135.364
134.772
132.296
130.996
130.059
129.952
129.705
129.170
128.389

168.633

0 ppm 50 100 150 200

Figure S3

Figure S4

Figure S5

Figure S6

Figure S7

KV (6.9.1)

Figure S8

Figure S9

KV-13 (3.11.01)

Figure S10

KV-13 (170-171) (3.11.01)

167.57

151.88
150.33

144.74

139.61
136.56
135.27
134.38
130.23
130.12
129.74
129.26
122.68

96.23

77.43
77.00
76.57

21.83
21.28

50

100

150

Figure S11

Figure S12

KV-2 (7.9.1)

Figure S13

Figure S14

Figure S15

KV-4 (18.3.2)

S16

Table 1. Crystal data and structure refinement for 4a.

Identification code	sv1	
Empirical formula	C ₂₁ H ₁₈ ClNO ₃ S	
Formula weight	399.87	
Temperature	293(2) K	
Diffractometer used	Siemens P4	
Radiation used, Wavelength	MoK α , 0.71073 Å	
Crystal system, Space group	Monoclinic, P2 ₁ /c	
Unit cell dimensions	a = 8.311(1) Å	$\alpha = 90^\circ$.
	b = 10.723(1) Å	$\beta = 92.25(1)^\circ$.
	c = 22.036(2) Å	$\gamma = 90^\circ$.
Volume	1962.3(3) Å ³	
Z, Calculated Density	4, 1.354 Mg/m ³	
Absorption coefficient	0.322 mm ⁻¹	
F(000)	832	
Crystal size	0.29 x 0.25 x 0.19 mm	
Theta range for data collection	1.85 to 24.50°.	
Scan type	2 θ - θ	
Scan speed	Variable, 2.0° to 60.0°/min. in ω	
Scan range (ω)	0.86° plus K α separation	
Background measurement	Stationary crystal and stationary counter at the beginning and end of scan, each for 25.0% of total scan time	
Index ranges	0 $\leq h \leq 9$, -12 $\leq k \leq 0$, -25 $\leq l \leq 25$	
Reflections collected	3497	
Independent reflections	3253 [R(int) = 0.0150]	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3253 / 0 / 245	
Goodness-of-fit on F ²	1.024	
Weighting scheme	1/[$\sigma^2(F_o^2) + (0.0447P)^2 + 0.47P$], P=(max(F _o ² , 0)+2*F _c ²)/3	
Data to parameter ratio	13.28:1	
Final R indices, 2572 reflections [I>2 σ (I)]	R1 = 0.0360, wR2 = 0.0917	
R indices (all data)	R1 = 0.0488, wR2 = 0.0996	
Extinction coefficient	0.0060(7)	
Largest diff. peak and hole	0.160 and -0.221 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for sv1. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Cl(1)	-3821(1)	8259(1)	-324(1)	121(1)
S(1)	4355(1)	9447(1)	2009(1)	52(1)
O(1)	5517(2)	8474(1)	1986(1)	64(1)
O(2)	4730(2)	10649(1)	1781(1)	66(1)
O(3)	1310(2)	10685(1)	1739(1)	67(1)
N(1)	2754(2)	8902(1)	1581(1)	49(1)
C(1)	3685(2)	9547(2)	2752(1)	49(1)
C(2)	3691(3)	8481(2)	3106(1)	58(1)
C(3)	3132(3)	8558(2)	3687(1)	69(1)
C(4)	2598(3)	9677(3)	3922(1)	70(1)
C(5)	2641(3)	10730(2)	3562(1)	66(1)
C(6)	3173(2)	10677(2)	2975(1)	55(1)
C(7)	1981(5)	9746(4)	4557(1)	118(1)
C(8)	2488(2)	7564(2)	1597(1)	45(1)
C(9)	1421(2)	7075(2)	1995(1)	54(1)
C(10)	1181(3)	5801(2)	2016(1)	61(1)
C(11)	1987(3)	4996(2)	1646(1)	57(1)
C(12)	3059(2)	5509(2)	1251(1)	57(1)
C(13)	3320(2)	6781(2)	1224(1)	50(1)
C(14)	1719(3)	3608(2)	1676(1)	87(1)
C(15)	1410(2)	9700(2)	1478(1)	52(1)
C(16)	148(2)	9273(2)	1027(1)	50(1)
C(17)	487(3)	8626(2)	502(1)	58(1)
C(18)	-732(3)	8326(2)	84(1)	66(1)
C(19)	-2279(3)	8659(2)	197(1)	69(1)
C(20)	-2648(3)	9307(2)	708(1)	72(1)
C(21)	-1428(3)	9616(2)	1123(1)	63(1)

Table 3. Bond lengths [Å] and angles [°] for sv1.

Cl(1)-C(19)	1.741(2)	S(1)-O(2)	1.4212(15)
S(1)-O(1)	1.4243(15)	S(1)-N(1)	1.7038(16)
S(1)-C(1)	1.754(2)	O(3)-C(15)	1.207(2)
N(1)-C(15)	1.418(3)	N(1)-C(8)	1.452(2)
C(1)-C(6)	1.381(3)	C(1)-C(2)	1.383(3)
C(2)-C(3)	1.383(3)	C(3)-C(4)	1.387(3)
C(4)-C(5)	1.382(3)	C(4)-C(7)	1.510(3)
C(5)-C(6)	1.385(3)	C(8)-C(9)	1.375(3)
C(8)-C(13)	1.381(3)	C(9)-C(10)	1.382(3)
C(10)-C(11)	1.378(3)	C(11)-C(12)	1.383(3)
C(11)-C(14)	1.507(3)	C(12)-C(13)	1.383(3)
C(15)-C(16)	1.489(3)	C(16)-C(21)	1.385(3)
C(16)-C(17)	1.386(3)	C(17)-C(18)	1.380(3)
C(18)-C(19)	1.367(3)	C(19)-C(20)	1.368(3)
C(20)-C(21)	1.377(3)		
O(2)-S(1)-O(1)	119.54(9)	O(2)-S(1)-N(1)	107.06(9)
O(1)-S(1)-N(1)	104.19(8)	O(2)-S(1)-C(1)	110.86(9)
O(1)-S(1)-C(1)	108.56(9)	N(1)-S(1)-C(1)	105.56(8)
C(15)-N(1)-C(8)	118.69(15)	C(15)-N(1)-S(1)	118.35(13)
C(8)-N(1)-S(1)	116.18(12)	C(6)-C(1)-C(2)	121.32(19)
C(6)-C(1)-S(1)	119.89(16)	C(2)-C(1)-S(1)	118.80(16)
C(1)-C(2)-C(3)	118.6(2)	C(2)-C(3)-C(4)	121.4(2)
C(5)-C(4)-C(3)	118.5(2)	C(5)-C(4)-C(7)	120.7(3)
C(3)-C(4)-C(7)	120.9(3)	C(4)-C(5)-C(6)	121.4(2)
C(1)-C(6)-C(5)	118.8(2)	C(9)-C(8)-C(13)	119.91(18)
C(9)-C(8)-N(1)	119.71(17)	C(13)-C(8)-N(1)	120.37(17)
C(8)-C(9)-C(10)	119.7(2)	C(11)-C(10)-C(9)	121.6(2)
C(10)-C(11)-C(12)	117.63(19)	C(10)-C(11)-C(14)	121.1(2)
C(12)-C(11)-C(14)	121.3(2)	C(13)-C(12)-C(11)	121.7(2)
C(8)-C(13)-C(12)	119.39(19)	O(3)-C(15)-N(1)	121.34(18)
O(3)-C(15)-C(16)	121.84(18)	N(1)-C(15)-C(16)	116.82(17)
C(21)-C(16)-C(17)	119.02(19)	C(21)-C(16)-C(15)	117.51(18)
C(17)-C(16)-C(15)	123.33(18)	C(18)-C(17)-C(16)	120.3(2)

C(19)-C(18)-C(17)	119.3(2)	C(18)-C(19)-C(20)	121.7(2)
C(18)-C(19)-Cl(1)	119.26(19)	C(20)-C(19)-Cl(1)	119.04(19)
C(19)-C(20)-C(21)	119.0(2)	C(20)-C(21)-C(16)	120.7(2)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for sv1. 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)	93(1)	171(1)	95(1)	-25(1)	-46(1)	11(1)
S(1)	48(1)	48(1)	60(1)	-4(1)	-1(1)	-6(1)
O(1)	46(1)	64(1)	82(1)	-12(1)	-1(1)	3(1)
O(2)	73(1)	54(1)	72(1)	0(1)	4(1)	-19(1)
O(3)	75(1)	49(1)	77(1)	-12(1)	-10(1)	9(1)
N(1)	50(1)	43(1)	54(1)	-3(1)	-2(1)	-1(1)
C(1)	45(1)	47(1)	55(1)	-4(1)	-9(1)	-2(1)
C(2)	59(1)	50(1)	65(1)	-2(1)	-12(1)	0(1)
C(3)	77(2)	69(2)	60(1)	11(1)	-11(1)	-10(1)
C(4)	69(2)	85(2)	56(1)	-7(1)	-5(1)	-10(1)
C(5)	62(1)	68(2)	65(1)	-18(1)	-6(1)	3(1)
C(6)	55(1)	49(1)	60(1)	-3(1)	-10(1)	-1(1)
C(7)	148(3)	138(3)	68(2)	-11(2)	23(2)	-17(2)
C(8)	46(1)	41(1)	47(1)	-1(1)	-5(1)	-1(1)
C(9)	50(1)	59(1)	53(1)	-3(1)	3(1)	-1(1)
C(10)	52(1)	67(1)	64(1)	13(1)	-2(1)	-15(1)
C(11)	52(1)	50(1)	67(1)	5(1)	-17(1)	-6(1)
C(12)	55(1)	49(1)	65(1)	-8(1)	-9(1)	7(1)
C(13)	49(1)	50(1)	51(1)	0(1)	1(1)	1(1)
C(14)	85(2)	53(1)	120(2)	8(1)	-19(2)	-12(1)
C(15)	57(1)	43(1)	54(1)	2(1)	2(1)	2(1)
C(16)	57(1)	46(1)	48(1)	5(1)	-2(1)	4(1)
C(17)	59(1)	58(1)	56(1)	2(1)	-4(1)	10(1)
C(18)	81(2)	66(1)	51(1)	-2(1)	-9(1)	11(1)
C(19)	67(2)	81(2)	56(1)	8(1)	-16(1)	2(1)
C(20)	55(1)	97(2)	63(1)	7(1)	-4(1)	9(1)
C(21)	63(1)	73(1)	53(1)	-1(1)	0(1)	9(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for sv1.

	x	y	z	U(eq)
H(2B)	4062	7728	2955	70
H(3A)	3115	7843	3926	83
H(5A)	2305	11489	3717	79
H(6A)	3185	11390	2734	66
H(7A)	2426	10322	4742	176
H(7B)	2210	8977	4766	176
H(7C)	839	9885	4536	176
H(9A)	863	7600	2249	65
H(10A)	457	5478	2286	73
H(12A)	3619	4984	998	68
H(13A)	4050	7105	956	60
H(14A)	1841	3371	2028	130
H(14B)	2478	3191	1429	130
H(14C)	644	3417	1529	130
H(17A)	1541	8394	431	70
H(18A)	-503	7902	-270	79
H(20A)	-3706	9535	775	86
H(21A)	-1666	10060	1470	75

Table 6. Torsion angles [°] for sv1.

O(2)-S(1)-N(1)-C(15)	-48.27(16)	O(1)-S(1)-N(1)-C(15)	-175.82(14)
C(1)-S(1)-N(1)-C(15)	69.90(16)	O(2)-S(1)-N(1)-C(8)	160.89(13)
O(1)-S(1)-N(1)-C(8)	33.35(16)	C(1)-S(1)-N(1)-C(8)	-80.93(15)
O(2)-S(1)-C(1)-C(6)	17.42(19)	O(1)-S(1)-C(1)-C(6)	150.61(15)
N(1)-S(1)-C(1)-C(6)	-98.17(16)	O(2)-S(1)-C(1)-C(2)	-162.29(15)
O(1)-S(1)-C(1)-C(2)	-29.10(18)	N(1)-S(1)-C(1)-C(2)	82.12(17)
C(6)-C(1)-C(2)-C(3)	1.6(3)	S(1)-C(1)-C(2)-C(3)	-178.73(16)
C(1)-C(2)-C(3)-C(4)	-1.0(3)	C(2)-C(3)-C(4)-C(5)	-0.3(3)
C(2)-C(3)-C(4)-C(7)	179.6(2)	C(3)-C(4)-C(5)-C(6)	1.2(3)
C(7)-C(4)-C(5)-C(6)	-178.7(2)	C(2)-C(1)-C(6)-C(5)	-0.7(3)
S(1)-C(1)-C(6)-C(5)	179.60(15)	C(4)-C(5)-C(6)-C(1)	-0.7(3)
C(15)-N(1)-C(8)-C(9)	-55.7(2)	S(1)-N(1)-C(8)-C(9)	95.05(18)
C(15)-N(1)-C(8)-C(13)	125.43(19)	S(1)-N(1)-C(8)-C(13)	-83.84(19)
C(13)-C(8)-C(9)-C(10)	-0.4(3)	N(1)-C(8)-C(9)-C(10)	-179.26(17)
C(8)-C(9)-C(10)-C(11)	0.0(3)	C(9)-C(10)-C(11)-C(12)	0.2(3)
C(9)-C(10)-C(11)-C(14)	179.8(2)	C(10)-C(11)-C(12)-C(13)	-0.1(3)
C(14)-C(11)-C(12)-C(13)	-179.6(2)	C(9)-C(8)-C(13)-C(12)	0.5(3)
N(1)-C(8)-C(13)-C(12)	179.38(17)	C(11)-C(12)-C(13)-C(8)	-0.3(3)
C(8)-N(1)-C(15)-O(3)	141.8(2)	S(1)-N(1)-C(15)-O(3)	-8.3(3)
C(8)-N(1)-C(15)-C(16)	-39.1(2)	S(1)-N(1)-C(15)-C(16)	170.82(13)
O(3)-C(15)-C(16)-C(21)	-32.9(3)	N(1)-C(15)-C(16)-C(21)	148.00(19)
O(3)-C(15)-C(16)-C(17)	142.8(2)	N(1)-C(15)-C(16)-C(17)	-36.3(3)
C(21)-C(16)-C(17)-C(18)	-0.3(3)	C(15)-C(16)-C(17)-C(18)	-175.92(19)
C(16)-C(17)-C(18)-C(19)	-0.8(3)	C(17)-C(18)-C(19)-C(20)	1.4(4)
C(17)-C(18)-C(19)-Cl(1)	-178.96(17)	C(18)-C(19)-C(20)-C(21)	-0.8(4)
Cl(1)-C(19)-C(20)-C(21)	179.54(19)	C(19)-C(20)-C(21)-C(16)	-0.3(4)
C(17)-C(16)-C(21)-C(20)	0.9(3)	C(15)-C(16)-C(21)-C(20)	176.8(2)