

SUPPLEMENTARY MATERIAL

A Simple Conversion of Creatinine to Creatol *via* Creatinine Chloroamine

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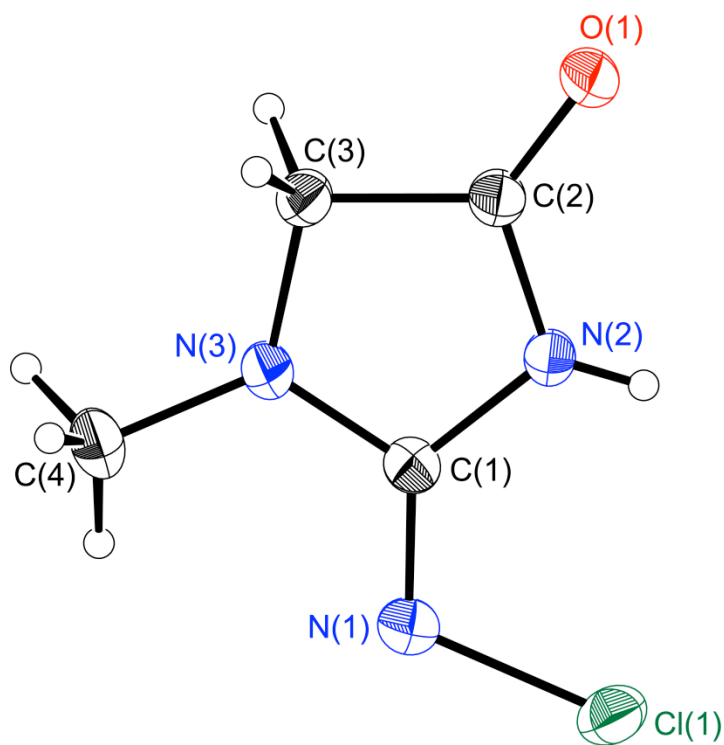
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Supporting Information for the X-ray structure
determination of **4a**
CCDC-1018085



EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₄ H ₆ ClN ₃ O
Formula Weight	147.56
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.400 X 0.250 X 0.200 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 6.7333(6) Å b = 7.1776(7) Å c = 7.7280(7) Å α = 86.686(3) °

$$\beta = 68.225(3)^\circ$$

$$\gamma = 65.585(3)^\circ$$

$$V = 313.80(5) \text{ \AA}^3$$

Space Group	P-1 (#2)
Z value	2
D _{calc}	1.562 g/cm ³
F ₀₀₀	152.00
μ(MoKa)	5.214 cm ⁻¹

B. Intensity Measurements

Diffractometer	SCX mini
Radiation	MoKa (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 26mA
Temperature	25.0°C
Detector Aperture	75 mm (diameter)
Data Images	540 exposures
ω oscillation Range	-120.0 - 60.0°
Exposure Rate	24.0 sec./°
Detector Swing Angle	-29.80°
ω oscillation Range	-120.0 - 60.0°
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ω oscillation Range	-120.0 - 60.0°
Exposure Rate	24.0 sec./°
Detector Swing Angle	-29.80°
Detector Position	49.70 mm
Pixel Size	0.146 mm

2 θ _{max}	54.9°
No. of Reflections Measured	Total: 3270 Unique: 1427 (R _{int} = 0.0389)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.810 - 0.901)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELX97)
Refinement	Full-matrix least-squares on F ²
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(F_o^2) + (0.0591 \cdot P)^2 + 0.0429 \cdot P]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$
2 θ _{max} cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1427
No. Variables	106
Reflection/Parameter Ratio	13.46
Residuals: R1 (I > 2.00 σ (I))	0.0398
Residuals: R (All reflections)	0.0458
Residuals: wR2 (All reflections)	0.1098
Goodness of Fit Indicator	1.067
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.28 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.24 e ⁻ /Å ³

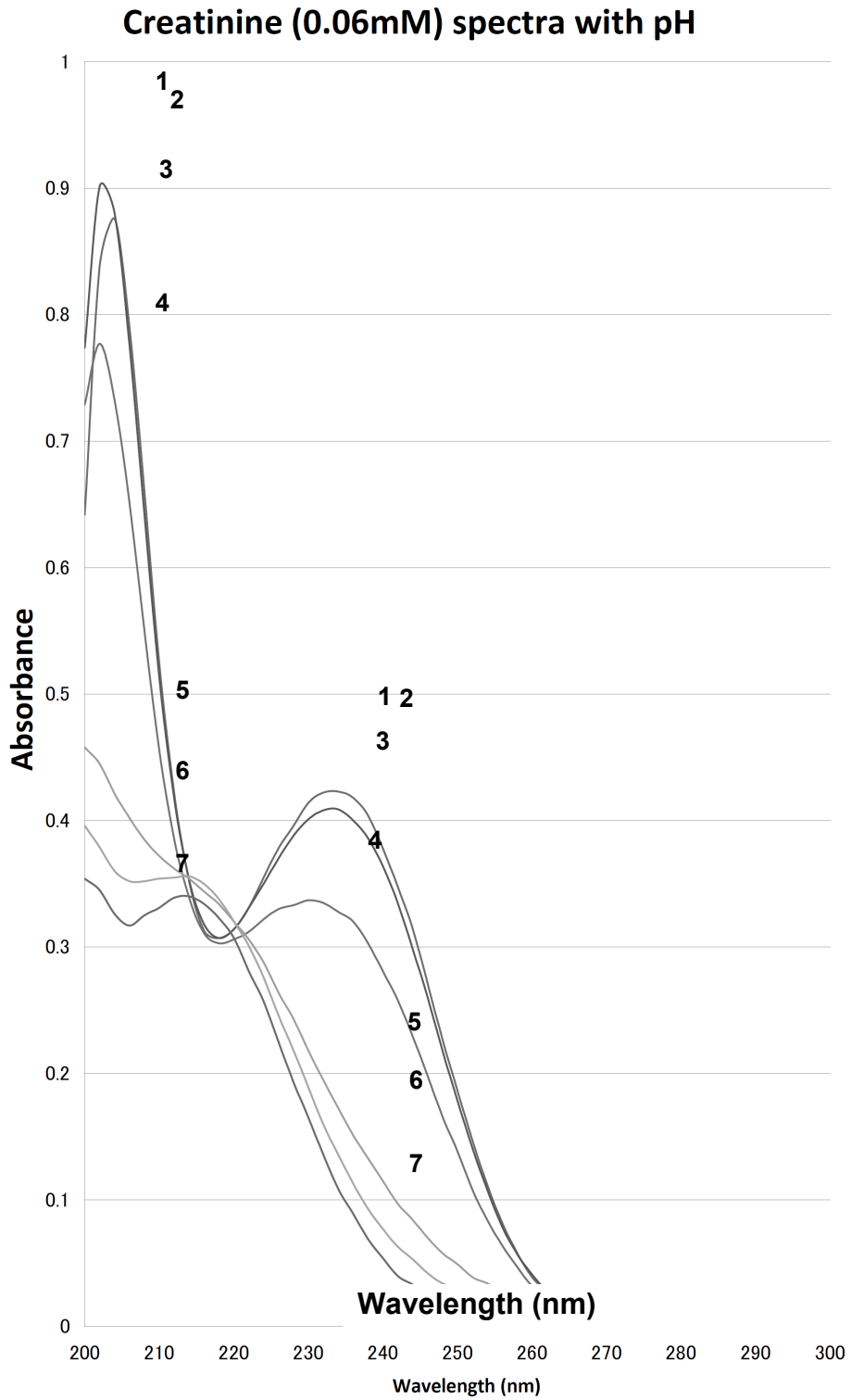
Bond lengths (Å)

atom	atom	distance	atom	atom	distance
C11	N1	1.7344(18)	O1	C2	1.221(3)
N1	C1	1.292(2)	N2	C1	1.387(3)
N2	C2	1.359(2)	N3	C1	1.354(3)
N3	C3	1.442(2)	N3	C4	1.445(4)
C2	C3	1.507(3)			

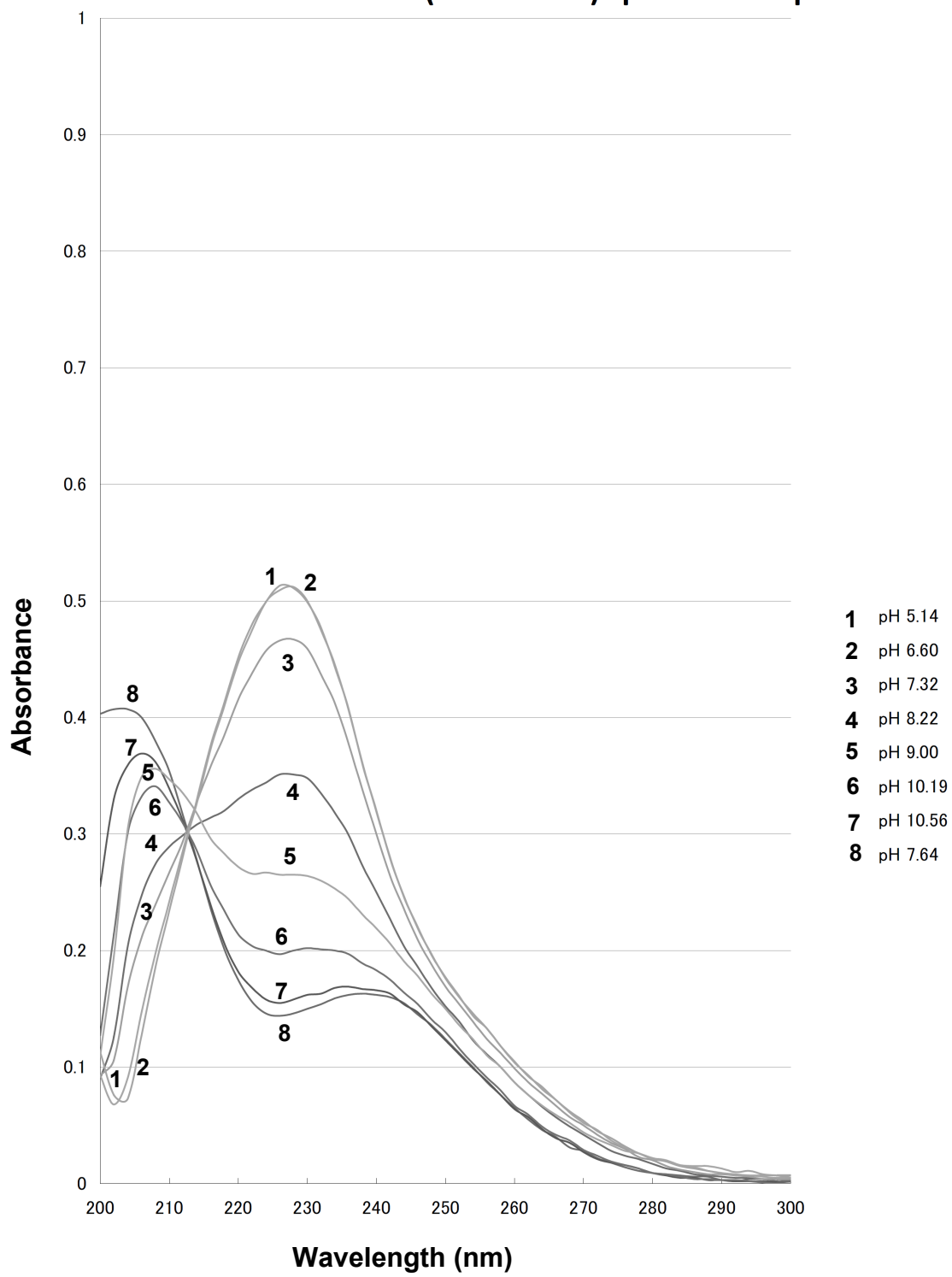
Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C11	N1	C1	112.38(14)	C1	N2	C2	111.52(16)
C1	N3	C3	111.47(15)	C1	N3	C4	123.35(17)
C3	N3	C4	123.66(17)	N1	C1	N2	130.22(17)
N1	C1	N3	122.17(17)	N2	C1	N3	107.61(13)
O1	C2	N2	126.14(18)	O1	C2	C3	126.97(16)
N2	C2	C3	106.89(15)	N3	C3	C2	102.30(14)

Spectroscopic determination of pKa's at 20°C.



Creatinine chloramine (0.0285mM) spectra with pH



pKa of creatinine

	0.314	0.3	0.219	0.064	0.027	0.01	0		
	100	95.5414	69.74522	20.38217	8.598726	3.184713	0		
pH	6.6	6.15	5.23	4.24	3.74	3.24	2.84		
234	0.423	0.409	0.328	0.173	0.136	0.119	0.109		
dl-d		0.014	0.095	0.25	0.287	0.304	0.314		
d-dM		0.3	0.219	0.064	0.027	0.01			
ratio		0.046667	0.43379	3.90625	10.62963	30.4			
log		-1.33099	-0.36272	0.59176	1.026518	1.482874			
		4.819007	4.867279	4.83176	4.766518	4.722874			

pKa= 4.801488

pKa of creatol

	0.124	0.122	0.112	0.097	0.056	0.031	0.015	0.002	0
	100	98.3871	90.32258	78.22581	45.16129	25	12.09677	1.612903	0
pH	5.91	5.55	5.05	4.7	4.12	3.71	3.35	2.71	2.19
d	0.162	0.16	0.15	0.135	0.094	0.069	0.053	0.04	0.038
dl-d		0.002	0.012	0.027	0.068	0.093	0.109	0.122	
d-dM		0.122	0.112	0.097	0.056	0.031	0.015	0.002	
ratio		0.016393	0.107143	0.278351	1.214286	3	7.266667	61	
log		-1.78533	-0.97004	-0.55541	0.084321	0.477121	0.861335	1.78533	
		3.76467	4.079963	4.144592	4.204321	4.187121	4.211335	4.49533	

pKa= 4.155333

pKa of creatinine-N-chloroamine

pH		5.14	6.6	7.32	7.64	8.22	9	10.56
	228	0.146	0.158	0.2	0.265	0.351	0.467	0.511
dl-d			0.353	0.311	0.246	0.16	0.044	
d-dM			0.012	0.054	0.119	0.205	0.321	
ratio			29.41667	5.759259	2.067227	0.780488	0.137072	
log			1.468593	0.760367	0.315388	-0.10763	-0.86305	
			8.068593	8.080367	7.955388	8.112366	8.136948	

pKa= 8.070732