SUUPLEMENTARY MATERIAL

PAPER CH14476. R1

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

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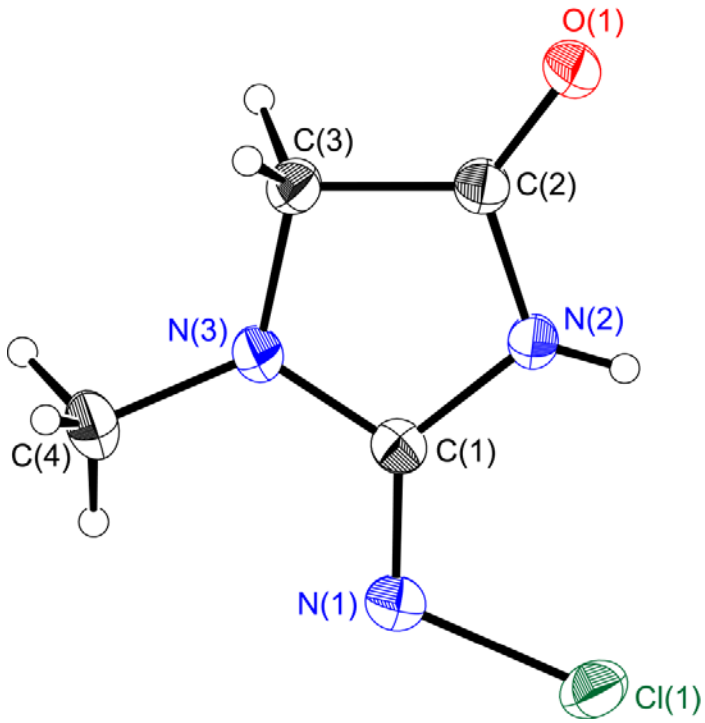
Supporting Information for the X-ray structure determination of **4a**

CCDC-1018085



EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_4H_6ClN_3O$
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) ^{o}

	β = 68.225(3) ° γ = 65.585(3) ° V = 313.80(5) Å^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 (l = 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°
Exposure Rate	24.0 sec./°
Detector Swing Angle	-29.80°
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

$2\theta_{max}$	54.9^{o}
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	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.0591 \cdot P)^2$ + $0.0429 \cdot P$] where P = $(Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	54.9^{o}
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1427
No. Variables	106
Reflection/Parameter Ratio	13.46
Residuals: R1 (I>2.00σ(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
Cl1	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
Cl1	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)

checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: I

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Bond precision:  C-C = 0.0030 A          Wavelength=0.71075

Cell:          a=6.7333(6)       b=7.1776(7)      c=7.7280(7)
               alpha=86.686(3)   beta=68.225(2)   gamma=65.585(2)
Temperature:   298 K

                Calculated                Reported
Volume          313.80(5)                 313.80(5)
Space group     P -1                      P -1
Hall group      -P 1                      -P 1
Moiety formula  C4 H6 Cl N3 O             C4 H6 Cl N3 O
Sum formula     C4 H6 Cl N3 O             C4 H6 Cl N3 O
Mr              147.57                    147.57
Dx,g cm-3       1.562                     1.562
Z               2                         2
Mu (mm-1)       0.522                     0.522
F000            152.0                     152.0
F000'           152.34
h,k,lmax        8,9,10                    8,9,10
Nref            1435                      1427
Tmin,Tmax       0.855,0.901               0.818,0.903
Tmin'           0.812

Correction method= NUMERICAL

Data completeness= 0.994          Theta(max)= 27.480

R(reflections)= 0.0398( 1256)     wR2(reflections)= 0.1098( 1427)

S = 1.067                 Npar= 106
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The following ALERTS were generated. Each ALERT has the format
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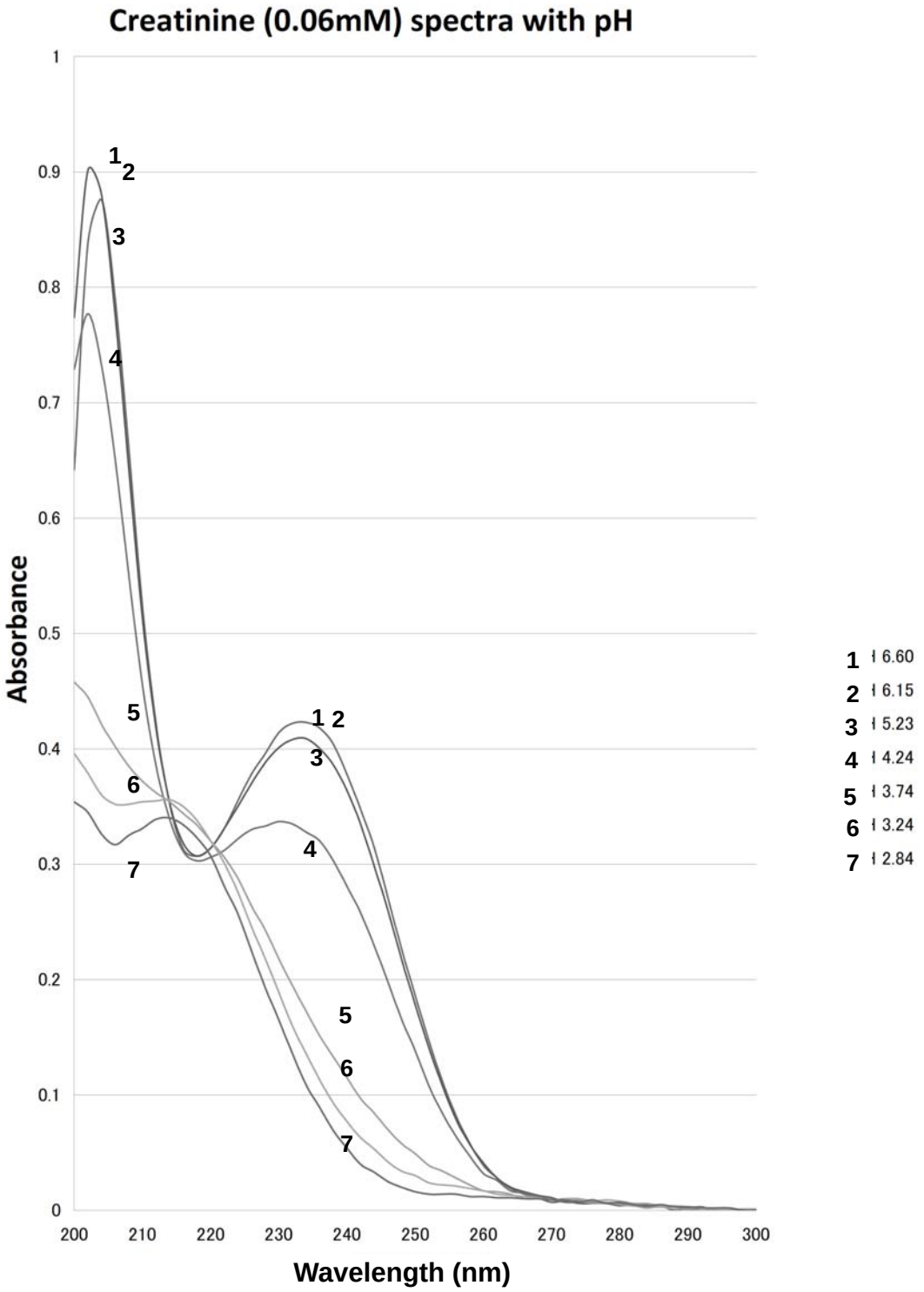
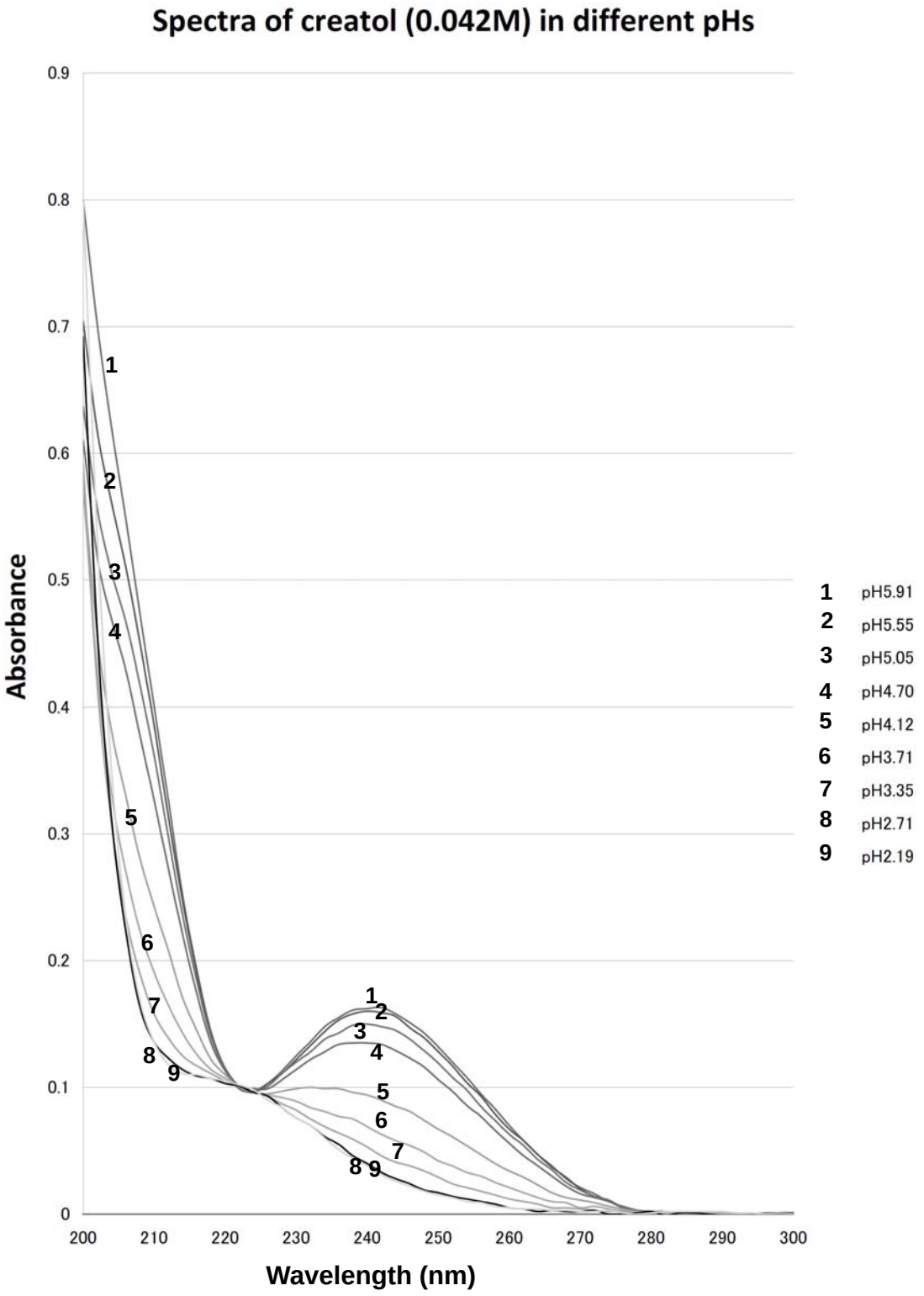
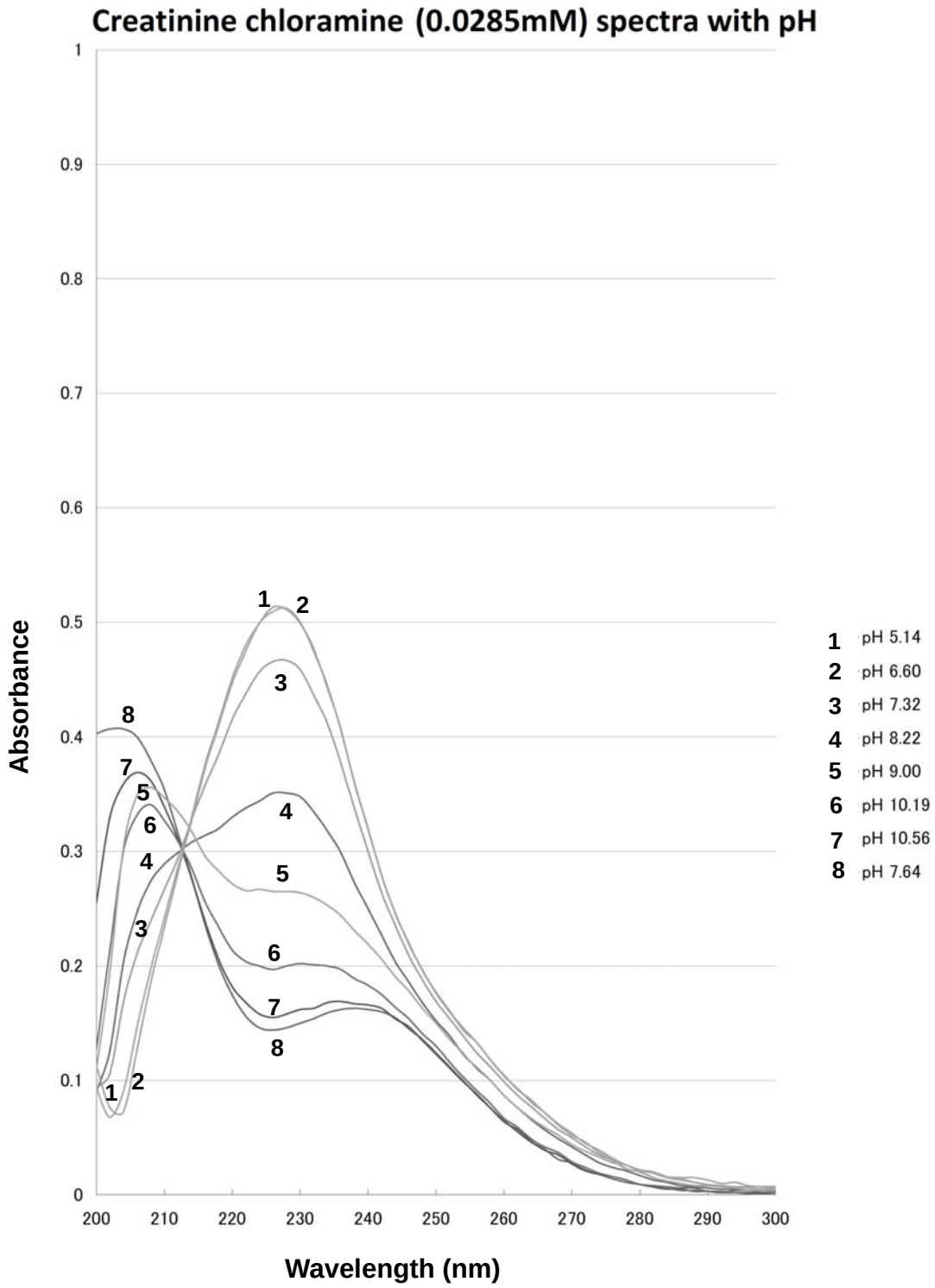
Click on the hyperlinks for more details of the test.

Spectroscopic determination of pKas at 20^{o}C.


Creatinine (0.06mM) spectra with pH
Absorbance
Wavelength (nm)
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
200
210
220
230
240
250
260
270
280
290
300
1 6.60
2 6.15
3 5.23
4 4.24
5 3.74
6 3.24
7 2.84


Spectra of creatol (0.042M) in different pHs
Absorbance
Wavelength (nm)
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
200
210
220
230
240
250
260
270
280
290
300
1 pH5.91
2 pH5.55
3 pH5.05
4 pH4.70
5 pH4.12
6 pH3.71
7 pH3.35
8 pH2.71
9 pH2.19


Creatinine chloramine (0.0285mM) spectra with pH
Absorbance
Wavelength (nm)
1 pH 5.14
2 pH 6.60
3 pH 7.32
4 pH 8.22
5 pH 9.00
6 pH 10.19
7 pH 10.56
8 pH 7.64

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		
dI-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
dI-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*-chloramine

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
dI-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