Crystal structure of *mer*-triaqua-[6-amino-5-formyl-1,3-dimethyluracilate benzoylhydrazone-*N,N',O*] chromium(II) nitrate, $C_{14}H_{20}CrN_7O_{12}$

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Abstract

$C_{14}H_{20}CrN_7O_{12}$, triclinic, $P\bar{1}$ (No. 2), $a = 7.000(1)$ Å, $b = 11.863(2)$ Å, $c = 13.031(2)$ Å, $\alpha = 104.69(2)^\circ$, $\beta = 93.53(2)^\circ$, $\gamma = 90.84(2)^\circ$, $V = 1044.3$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.051$, $wR_{\text{ref}}(F^2) = 0.135$, $T = 293$ K.

Source of material

The title compound was synthesized by mixing equimolecular amounts of the benzoylhydrazone ligand and $Cr(NO_3)_3 \cdot 9H_2O$ in methanol. The resulting red solution was stirred, heated during one hour and left to evaporate at room temperature. One day later, orange crystals were isolated [1].

Discussion

The crystal structure is made up by monomeric octahedral $[Cr(HBEZDO)(H_2O)_3]^{2+}$ units plus uncoordinated nitrate anions, where HBEZDO[−] represents the deprotonated form of 6-amino-5-formyl-1,3-dimethyluracil benzoylhydrazone. The organic ligand binds tridentately through the N6, N51 and O52 giving rise to two adjacent five- and six-membered chelate rings. The assignment of the N6 as metal-ligand binding site, which could be interchanged with the O4 due to the pseudosymmetry of the uracil ring through the O2–C2–C5–C51 pseudo-two-fold axis, has been made from the presence of the remaining hydrogen atom on N6, clearly seen in the difference maps. The bond angles are near

the ideal values of 90° and 180° with the larger exceptions of N6–Cr–O52 ($168.2(1)^\circ$) and N51–Cr–O52 ($80.3(1)^\circ$) [2–6]. The HBEZDO[−] ligand is practically planar, the dihedral angle defined by the mean planes passing through the endocyclic atoms of the uracil ring and the benzene one being 2.0° , whereas in the free ligand both rings are twisted by 38.2° [1]. The planarity of the ligand may be explained by the steric requirements of the chelate rings and also explains the meridional arrangement of the three coordinated water molecules. The molecular units are extensively 2-D hydrogen-bonded.

Table 1. Data collection and handling.

Crystal:	orange, plate-like, size $0.16 \times 0.16 \times 0.28$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	6.28 cm^{-1}
Diffractometer, scan mode:	STOE-IPDS, 128 exposures, $\Delta\varphi = 1.6^\circ$
$2\theta_{\text{max}}$:	52.14°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	9075, 3801
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2552
$N(\text{param})_{\text{refined}}$:	361
Programs:	XTM-93 [7], SHELXL-97 [8], PLUTON-95 [9], PARST [10]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(6)	2i	0.5764	0.6107	0.8710	0.018
H(52)	2i	0.7550	0.4725	0.4491	0.032
H(5B)	2i	0.766(7)	0.182(4)	0.167(3)	0.047
H(51)	2i	0.812(6)	0.657(3)	0.556(3)	0.031
H(2B)	2i	0.601(7)	0.126(4)	0.484(3)	0.043
H(4B)	2i	0.649(7)	0.000(4)	0.184(4)	0.052
H(3B)	2i	0.565(7)	−0.026(4)	0.344(3)	0.051
H(6B)	2i	0.772(7)	0.332(4)	0.314(3)	0.039
H(31)	2i	0.761(9)	1.074(4)	0.769(4)	0.065
H(32)	2i	0.962(9)	1.058(5)	0.793(4)	0.065
H(33)	2i	0.846(8)	1.103(5)	0.880(4)	0.065
H(11)	2i	0.646(9)	0.692(5)	0.997(4)	0.080
H(12)	2i	0.76(1)	0.792(5)	1.043(5)	0.080
H(13)	2i	0.56(1)	0.803(6)	1.015(5)	0.080
H(1W1)	2i	0.299(7)	0.405(4)	0.651(3)	0.039
H(2W1)	2i	0.333(8)	0.486(5)	0.635(4)	0.039
H(1W2)	2i	0.441(8)	0.405(4)	0.844(4)	0.046
H(2W2)	2i	0.605(8)	0.364(4)	0.861(4)	0.046
H(1W3)	2i	0.941(8)	0.382(5)	0.749(4)	0.053
H(2W3)	2i	0.962(8)	0.492(5)	0.806(4)	0.053

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cr	2i	0.6325(1)	0.45541(5)	0.71549(4)	0.0381(4)	0.0204(3)	0.0154(3)	−0.0047(2)	0.0017(2)	0.0017(2)
N(6)	2i	0.6318(4)	0.6088(2)	0.8135(2)	0.028(2)	0.010(1)	0.007(1)	−0.007(1)	0.007(1)	0.0001(9)
O(52)	2i	0.6626(4)	0.3193(2)	0.6007(2)	0.049(2)	0.022(1)	0.020(1)	−0.003(1)	0.005(1)	0.0029(9)
N(51)	2i	0.7187(5)	0.5309(2)	0.6065(2)	0.035(2)	0.021(2)	0.016(1)	−0.004(1)	0.002(1)	0.000(1)
N(52)	2i	0.7304(5)	0.4529(2)	0.5062(2)	0.041(2)	0.023(2)	0.012(1)	−0.003(1)	0.005(1)	−0.001(1)
O(1A)	2i	1.2464(6)	0.5085(3)	0.9503(3)	0.087(3)	0.041(2)	0.055(2)	0.021(2)	0.010(2)	0.011(2)
O(1B)	2i	1.0763(6)	0.2401(3)	0.7352(2)	0.111(3)	0.036(2)	0.026(1)	−0.003(2)	0.012(2)	0.007(1)
O(3B)	2i	1.0218(6)	0.1026(2)	0.5926(2)	0.100(3)	0.027(2)	0.040(2)	−0.019(2)	0.007(2)	0.004(1)
O(4)	2i	0.8641(5)	0.8820(3)	0.6319(2)	0.063(3)	0.041(2)	0.042(2)	−0.006(2)	0.011(2)	0.010(1)
N(1A)	2i	1.1827(6)	0.6066(3)	0.9616(3)	0.044(3)	0.038(2)	0.043(2)	−0.004(2)	−0.002(2)	0.011(2)
N(2B)	2i	1.0821(6)	0.2006(3)	0.6377(2)	0.055(3)	0.026(2)	0.028(2)	0.002(2)	0.007(2)	0.005(1)
N(1)	2i	0.7108(5)	0.7928(3)	0.8993(2)	0.048(2)	0.024(2)	0.019(1)	−0.009(1)	0.005(1)	0.002(1)
O(2)	2i	0.7644(6)	0.9824(2)	0.9868(2)	0.101(3)	0.030(2)	0.026(1)	−0.016(2)	0.020(2)	−0.010(1)
O(2B)	2i	1.1508(6)	0.2643(3)	0.5846(2)	0.096(3)	0.045(2)	0.030(1)	−0.034(2)	0.010(2)	0.009(1)
O(2A)	2i	1.2288(6)	0.6860(3)	1.0425(2)	0.083(3)	0.043(2)	0.048(2)	−0.002(2)	−0.020(2)	0.000(2)
C(5B)	2i	0.7244(7)	0.1701(4)	0.2278(3)	0.041(3)	0.047(3)	0.025(2)	0.010(2)	0.006(2)	−0.002(2)
C(51)	2i	0.7651(6)	0.6400(3)	0.6166(3)	0.034(3)	0.022(2)	0.018(2)	−0.005(2)	0.004(2)	0.003(1)
N(3)	2i	0.8139(5)	0.9307(2)	0.8107(2)	0.041(2)	0.016(2)	0.026(1)	−0.006(1)	0.006(1)	−0.002(1)
C(2B)	2i	0.6359(7)	0.1364(3)	0.4223(3)	0.048(3)	0.026(2)	0.029(2)	0.000(2)	0.006(2)	−0.001(2)
C(6)	2i	0.6981(6)	0.7060(3)	0.8076(2)	0.032(3)	0.029(2)	0.015(2)	−0.001(2)	0.003(1)	−0.001(1)
C(4B)	2i	0.6649(7)	0.0618(4)	0.2358(3)	0.050(3)	0.039(2)	0.028(2)	0.010(2)	−0.001(2)	−0.015(2)
O(3A)	2i	1.0739(6)	0.6298(3)	0.8913(3)	0.074(3)	0.051(2)	0.067(2)	−0.008(2)	−0.036(2)	0.021(2)
C(3B)	2i	0.6219(7)	0.0441(4)	0.3332(3)	0.046(3)	0.026(2)	0.049(2)	−0.003(2)	0.000(2)	−0.002(2)
C(52)	2i	0.6983(6)	0.3452(3)	0.5126(2)	0.029(3)	0.023(2)	0.021(2)	−0.001(2)	0.000(1)	−0.000(1)
C(6B)	2i	0.7418(6)	0.2629(4)	0.3171(3)	0.037(3)	0.033(2)	0.025(2)	0.007(2)	0.006(2)	0.000(2)
C(2)	2i	0.7638(7)	0.9076(3)	0.9051(3)	0.048(3)	0.027(2)	0.026(2)	−0.007(2)	0.009(2)	0.001(2)
C(1B)	2i	0.6960(6)	0.2477(3)	0.4157(3)	0.032(3)	0.024(2)	0.026(2)	0.002(2)	0.001(2)	−0.002(1)
C(3)	2i	0.8617(9)	1.0538(4)	0.8158(4)	0.067(4)	0.020(2)	0.039(2)	−0.007(2)	0.014(2)	−0.001(2)
C(5)	2i	0.7590(6)	0.7302(3)	0.7131(2)	0.033(3)	0.019(2)	0.020(2)	−0.004(1)	0.005(1)	0.001(1)
C(4)	2i	0.8144(6)	0.8476(3)	0.7165(2)	0.028(3)	0.024(2)	0.021(2)	−0.003(2)	0.003(1)	0.002(1)
C(1)	2i	0.664(1)	0.7668(4)	0.9997(3)	0.100(5)	0.035(3)	0.020(2)	−0.017(3)	0.018(2)	−0.003(2)
OW(1)	2i	0.3601(5)	0.4622(3)	0.6663(2)	0.041(2)	0.028(2)	0.027(1)	−0.010(1)	−0.003(1)	0.010(1)
OW(2)	2i	0.5267(6)	0.3762(3)	0.8199(2)	0.052(3)	0.038(2)	0.027(2)	−0.002(2)	0.004(1)	0.013(1)
OW(3)	2i	0.8953(5)	0.4426(3)	0.7712(2)	0.051(2)	0.034(2)	0.038(2)	0.004(2)	−0.008(1)	−0.005(1)

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