Structure Determination of ZnNDC

A colorless, block crystal (0.20 x 0.14 x 0.14 mm) of $Zn(NDC)_{1.5}$(Triethyl ammonium)(Chlorobenzne)(N,N'-Diethyformamide) was coated with a light hydrocarbon-based inert oil and mounted on a standard Siemens SMART CCD-based X-ray diffractometor equipped with a normal focus Mo-target X-ray tube ($\lambda$ = 0.71073 Å) operated at 2000 W power (50 kV, 40 mA). The X-ray intensities were measured at 158(2) K; the detector was placed at a distance of 4.916 cm from the crystal. A total of 2292 frames were collected with a scan width of 0.3° in $\omega$ and phi with an exposure time of 30 s/frame. The frames were integrated with the Siemens SAINT software package with a narrow frame algorithm. The integration of the data using triclinic unit cell yielded a total of 12232 reflections to a maximum $2\theta$ value of 46.66° of which 4859 were independent and 3271 were greater than $2\sigma(I)$. The final cell constants (Table 1) were based on xyz centroids of 3314 reflections above 10 $\sigma(I)$. Analysis of the data showed negligible decay during data collection collection; the data were corrected for absorption using an empirical method (SADABS) with transmission coefficients ranging from 0.692 to 0.914. The structure was solved and refined with the Siemens SHELXTL (version 5.10) software package, using the centrosymmetric space group $P(-1)$ with $Z = 2$ for the formula. All non-hydrogen atoms were refined anisotropically with the hydrogen atoms atoms except for the water molecule placed in idealized positions; the hydrogen atom of the triethylammonium ion was found in the difference Fourier map and their positional parameters were refined. Refinement was carried out using full matrix least-squares. The final refinement based on $F^2$ converged to R1(obs) = 0.0588 and wR2 = 0.1565 (all data). Additional details are presented in Table 1 and are given as Supporting information.

Sheldrick, G. M. SHELXTL, v. 5.10; Bruker Analytical X-ray, Madison, WI, 1997.

Sheldrick, G. M. SADABS. Program for Empirical Absorption Correction of Area Detector Data, University of Göttingen: Gottingen, Germany, 1996.

Saint Plus, v. 6.01, Bruker Analytical X-ray, Madison, WI, 1999.

Table 1. Crystal data and structure refinement for ZnNDC.

| | | |
|---|---|---|
| Identification code | ZnNDC | |
| Empirical formula | C35 H41 Cl N2 O7 Zn | |
| Formula weight | 702.52 | |
| Temperature | 158(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Triclinic | |
| Space group | P(-1) | |
| Unit cell dimensions | a = 9.9518(8) Å | α= 72.3770(10)°. |
| | b = 11.5761(9) Å | β= 83.1650(10)°. |
| | c = 15.5562(12) Å | γ = 84.3260(10)°. |
| Volume | 1692.1(2) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.379 $Mg/m^3$ | |
| Absorption coefficient | 0.855 $mm^{-1}$ | |
| F(000) | 736 | |
| Crystal size | .20 x .14 x .14 $mm^3$ | |
| Theta range for data collection | 1.38 to 23.33°. | |
| Index ranges | -11<=h<=11, -12<=k<=12, -17<=l<=17 | |
| Reflections collected | 12232 | |
| Independent reflections | 4859 [R(int) = 0.0541] | |
| Completeness to theta = 23.33° | 99.2 % | |
| Absorption correction | SADABS | |
| Max. and min. transmission | .914 and .692 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4859 / 0 / 418 | |
| Goodness-of-fit on $F^2$ | 0.987 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0588, wR2 = 0.1426 | |
| R indices (all data) | R1 = 0.0949, wR2 = 0.1565 | |
| Largest diff. peak and hole | 0.967 and -0.611 $e.Å^{-3}$ | |

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Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for ZnNDC. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Zn(1) | 4146(1) | 927(1) | 8878(1) | 28(1) |
| Cl(1) | 5097(2) | 12046(2) | 3389(2) | 106(1) |
| O(1) | 2867(3) | 1906(3) | 9439(2) | 32(1) |
| O(2) | 973(3) | 1275(3) | 9131(3) | 37(1) |
| O(3) | 4210(3) | -814(3) | 9025(2) | 33(1) |
| O(4) | 4197(3) | -1049(3) | 10499(2) | 31(1) |
| O(5) | 4782(4) | 1935(3) | 7675(3) | 37(1) |
| O(6) | 2742(4) | 1737(4) | 7294(3) | 46(1) |
| O(7) | -347(4) | 6673(4) | 5282(3) | 65(1) |
| C(1) | 1571(5) | 1976(5) | 9384(3) | 28(1) |
| C(2) | 800(5) | 3023(5) | 9650(3) | 28(1) |
| C(3) | 1447(5) | 3919(5) | 9793(3) | 30(1) |
| C(4) | 720(5) | 4961(5) | 9979(4) | 28(1) |
| C(5) | 1370(5) | 5915(5) | 10123(4) | 36(1) |
| C(6) | -640(5) | 3121(5) | 9701(4) | 36(1) |
| C(7) | 4307(5) | -1464(5) | 9842(4) | 29(1) |
| C(8) | 4546(5) | -2829(5) | 10023(4) | 39(2) |
| C(9) | 4805(5) | -3334(5) | 9333(5) | 44(2) |
| C(10) | 5039(5) | -4667(5) | 9542(4) | 35(1) |
| C(11) | 5299(6) | -5170(6) | 8844(5) | 48(2) |
| C(12) | 5495(5) | -6421(5) | 9057(5) | 44(2) |
| C(13) | 3852(6) | 2215(5) | 7143(4) | 37(1) |
| C(14) | 4111(5) | 3210(5) | 6271(4) | 34(1) |
| C(15) | 3245(6) | 3448(5) | 5576(4) | 39(1) |
| C(16) | 3452(6) | 4375(5) | 4788(4) | 41(2) |
| C(17) | 4557(5) | 5119(5) | 4658(4) | 34(1) |
| C(18) | 4802(5) | 6083(5) | 3843(4) | 36(1) |
| C(19) | 3980(7) | 10909(8) | 3974(5) | 71(2) |
| C(20) | 4337(9) | 9749(9) | 3995(6) | 85(3) |
| C(21) | 3425(11) | 8891(9) | 4467(7) | 100(3) |
| C(22) | 2129(8) | 9121(8) | 4919(5) | 86(3) |

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| | | | | |
|---|---|---|---|---|
| C(23) | 1874(8) | 10382(8) | 4815(5) | 74(2) |
| C(24) | 2761(7) | 11267(7) | 4369(5) | 62(2) |
| N(1) | -13(5) | 670(4) | 7731(3) | 34(1) |
| N(2) | 401(4) | 5795(4) | 6676(3) | 37(1) |
| C(7X) | 2529(6) | 5245(6) | 7441(5) | 58(2) |
| C(8X) | 1257(6) | 4822(5) | 7250(4) | 45(2) |
| C(9X) | -394(6) | 6649(5) | 7088(4) | 48(2) |
| C(10X) | -1772(6) | 6189(6) | 7523(4) | 55(2) |
| C(11X) | 341(6) | 5881(6) | 5809(4) | 48(2) |
| C(1X) | 1527(6) | -1228(6) | 7970(5) | 65(2) |
| C(2X) | 834(6) | -196(6) | 7308(4) | 46(2) |
| C(3X) | -2166(6) | -452(6) | 8081(5) | 53(2) |
| C(4X) | -1114(6) | 78(5) | 8443(4) | 42(2) |
| C(5X) | -529(6) | 1743(5) | 7007(4) | 48(2) |
| C(6X) | -1375(7) | 2677(6) | 7373(5) | 71(2) |

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

| | | | |
|---|---|---|---|
| Zn(1)-O(1) | 1.928(3) | Zn(1)-O(5) | 1.951(4) |
| Zn(1)-O(3) | 1.954(3) | Zn(1)-O(4)#1 | 2.047(4) |
| Cl(1)-C(19) | 1.766(9) | O(1)-C(1) | 1.295(6) |
| O(2)-C(1) | 1.231(6) | O(3)-C(7) | 1.275(6) |
| O(4)-C(7) | 1.244(6) | O(4)-Zn(1)#1 | 2.047(3) |
| O(5)-C(13) | 1.268(6) | O(6)-C(13) | 1.249(7) |
| O(7)-C(11X) | 1.239(7) | C(1)-C(2) | 1.507(7) |
| C(2)-C(3) | 1.359(7) | C(2)-C(6) | 1.421(7) |
| C(3)-C(4) | 1.433(7) | C(4)-C(5) | 1.419(7) |
| C(4)-C(4)#2 | 1.422(9) | C(5)-C(6)#2 | 1.351(7) |
| C(6)-C(5)#2 | 1.351(7) | C(7)-C(8) | 1.518(7) |
| C(8)-C(9) | 1.358(8) | C(8)-C(12)#3 | 1.430(8) |
| C(9)-C(10) | 1.478(8) | C(11)-C(10) | 1.367(8) |
| C(11)-C(12) | 1.384(8) | C(10)-C(10)#3 | 1.398(11) |
| C(12)-C(8)#3 | 1.430(8) | C(13)-C(14) | 1.504(7) |
| C(14)-C(18)#4 | 1.383(8) | C(14)-C(15) | 1.408(7) |
| C(15)-C(16) | 1.372(8) | C(16)-C(17) | 1.422(8) |
| C(17)-C(17)#4 | 1.409(10) | C(17)-C(18) | 1.426(7) |
| C(18)-C(14)#4 | 1.383(8) | C(19)-C(20) | 1.345(10) |
| C(19)-C(24) | 1.378(9) | C(20)-C(21) | 1.389(13) |
| C(21)-C(22) | 1.434(12) | C(22)-C(23) | 1.419(11) |
| C(23)-C(24) | 1.383(11) | N(1)-C(5X) | 1.496(7) |
| N(1)-C(2X) | 1.499(7) | N(1)-C(4X) | 1.515(7) |
| N(2)-C(11X) | 1.329(7) | N(2)-C(9X) | 1.457(7) |
| N(2)-C(8X) | 1.474(7) | C(7X)-C(8X) | 1.494(8) |
| C(9X)-C(10X) | 1.525(8) | C(1X)-C(2X) | 1.492(8) |
| C(3X)-C(4X) | 1.505(8) | C(5X)-C(6X) | 1.510(9) |
| | | | |
| O(1)-Zn(1)-O(5) | 108.28(15) | O(1)-Zn(1)-O(3) | 129.32(15) |
| O(5)-Zn(1)-O(3) | 115.36(16) | O(1)-Zn(1)-O(4)#1 | 97.73(14) |
| O(5)-Zn(1)-O(4)#1 | 96.31(15) | O(3)-Zn(1)-O(4)#1 | 101.73(14) |
| C(1)-O(1)-Zn(1) | 123.4(3) | C(7)-O(3)-Zn(1) | 113.1(3) |
| C(7)-O(4)-Zn(1)#1 | 121.5(3) | C(13)-O(5)-Zn(1) | 111.9(3) |
| O(2)-C(1)-O(1) | 124.9(5) | O(2)-C(1)-C(2) | 120.4(5) |

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| | | | |
|---|---|---|---|
| O(1)-C(1)-C(2) | 114.7(5) | C(3)-C(2)-C(6) | 118.7(5) |
| C(3)-C(2)-C(1) | 121.7(5) | C(6)-C(2)-C(1) | 119.5(5) |
| C(2)-C(3)-C(4) | 121.9(5) | C(5)-C(4)-C(4)#2 | 118.7(6) |
| C(5)-C(4)-C(3) | 123.1(4) | C(4)#2-C(4)-C(3) | 118.2(6) |
| C(6)#2-C(5)-C(4) | 120.9(5) | C(5)#2-C(6)-C(2) | 121.6(5) |
| O(4)-C(7)-O(3) | 123.9(5) | O(4)-C(7)-C(8) | 118.2(5) |
| O(3)-C(7)-C(8) | 117.9(5) | C(9)-C(8)-C(12)#3 | 120.3(5) |
| C(9)-C(8)-C(7) | 121.3(6) | C(12)#3-C(8)-C(7) | 118.4(5) |
| C(8)-C(9)-C(10) | 119.4(6) | C(10)-C(11)-C(12) | 118.0(6) |
| C(11)-C(10)-C(10)#3 | 124.3(7) | C(11)-C(10)-C(9) | 119.1(6) |
| C(10)#3-C(10)-C(9) | 116.6(6) | C(11)-C(12)-C(8)#3 | 121.4(6) |
| O(6)-C(13)-O(5) | 125.0(5) | O(6)-C(13)-C(14) | 118.1(5) |
| O(5)-C(13)-C(14) | 116.9(5) | C(18)#4-C(14)-C(15) | 119.5(5) |
| C(18)#4-C(14)-C(13) | 119.7(5) | C(15)-C(14)-C(13) | 120.9(5) |
| C(16)-C(15)-C(14) | 121.3(5) | C(15)-C(16)-C(17) | 120.3(5) |
| C(17)#4-C(17)-C(16) | 119.1(6) | C(17)#4-C(17)-C(18) | 119.4(6) |
| C(16)-C(17)-C(18) | 121.5(5) | C(14)#4-C(18)-C(17) | 120.5(5) |
| C(20)-C(19)-C(24) | 123.3(9) | C(20)-C(19)-Cl(1) | 119.1(7) |
| C(24)-C(19)-Cl(1) | 117.7(7) | C(19)-C(20)-C(21) | 116.7(8) |
| C(20)-C(21)-C(22) | 126.5(8) | C(23)-C(22)-C(21) | 110.4(9) |
| C(24)-C(23)-C(22) | 125.3(8) | C(19)-C(24)-C(23) | 117.8(7) |
| C(5X)-N(1)-C(2X) | 109.9(5) | C(5X)-N(1)-C(4X) | 113.5(4) |
| C(2X)-N(1)-C(4X) | 113.8(4) | C(11X)-N(2)-C(9X) | 120.9(5) |
| C(11X)-N(2)-C(8X) | 121.0(5) | C(9X)-N(2)-C(8X) | 118.2(5) |
| N(2)-C(8X)-C(7X) | 113.8(5) | N(2)-C(9X)-C(10X) | 112.0(5) |
| O(7)-C(11X)-N(2) | 124.9(6) | C(1X)-C(2X)-N(1) | 113.9(5) |
| C(3X)-C(4X)-N(1) | 114.0(5) | N(1)-C(5X)-C(6X) | 113.5(5) |

Symmetry transformations used to generate equivalent atoms:

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

#4 -x+1,-y+1,-z+1

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Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for ZnNDC. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Zn(1) | 26(1) | 27(1) | 31(1) | -7(1) | 4(1) | -6(1) |
| Cl(1) | 72(1) | 137(2) | 95(2) | -16(2) | 5(1) | -13(1) |
| O(1) | 25(2) | 30(2) | 44(2) | -14(2) | -1(2) | -4(2) |
| O(2) | 32(2) | 38(2) | 46(3) | -22(2) | 5(2) | -12(2) |
| O(3) | 37(2) | 32(2) | 31(2) | -11(2) | -2(2) | -6(2) |
| O(4) | 28(2) | 32(2) | 32(2) | -9(2) | 1(2) | -6(2) |
| O(5) | 38(2) | 36(2) | 33(2) | -2(2) | -4(2) | -6(2) |
| O(6) | 42(2) | 48(3) | 40(3) | 1(2) | 6(2) | -21(2) |
| O(7) | 59(3) | 75(3) | 53(3) | -10(3) | -17(2) | 12(3) |
| C(1) | 33(3) | 31(3) | 18(3) | -5(2) | 4(2) | -8(3) |
| C(2) | 25(3) | 27(3) | 30(3) | -5(2) | 1(2) | -5(2) |
| C(3) | 22(3) | 31(3) | 34(3) | -8(3) | 6(2) | -4(2) |
| C(4) | 21(2) | 32(3) | 32(3) | -11(3) | 0(2) | -8(2) |
| C(5) | 18(3) | 41(4) | 51(4) | -18(3) | 9(3) | -9(3) |
| C(6) | 27(3) | 36(3) | 50(4) | -21(3) | 3(3) | -12(3) |
| C(7) | 15(3) | 27(3) | 46(4) | -15(3) | 1(3) | -6(2) |
| C(8) | 17(3) | 36(3) | 70(5) | -25(3) | -1(3) | -7(2) |
| C(9) | 23(3) | 38(4) | 72(5) | -19(3) | 2(3) | -10(3) |
| C(11) | 37(3) | 50(4) | 51(4) | -8(3) | 2(3) | -9(3) |
| C(10) | 17(3) | 58(4) | 34(3) | -21(3) | 6(3) | -6(3) |
| C(12) | 30(3) | 27(3) | 69(5) | -4(3) | -3(3) | -6(3) |
| C(13) | 50(4) | 28(3) | 29(4) | -7(3) | 9(3) | -5(3) |
| C(14) | 38(3) | 31(3) | 29(3) | -5(3) | 4(3) | -4(3) |
| C(15) | 38(3) | 35(3) | 39(4) | -4(3) | 2(3) | -10(3) |
| C(16) | 36(3) | 50(4) | 36(4) | -7(3) | -2(3) | -13(3) |
| C(17) | 32(3) | 35(3) | 34(4) | -6(3) | 0(2) | -8(3) |
| C(18) | 37(3) | 33(3) | 36(4) | -8(3) | 1(3) | -5(3) |
| C(19) | 65(5) | 95(6) | 57(5) | -28(5) | -9(4) | -1(5) |
| C(20) | 83(6) | 94(7) | 100(7) | -69(6) | -22(5) | 39(6) |
| C(21) | 112(8) | 75(6) | 136(9) | -59(7) | -50(7) | 25(6) |
| C(22) | 82(6) | 110(7) | 86(6) | -63(6) | -43(5) | 57(5) |

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| | | | | | | |
|---|---|---|---|---|---|---|
| C(23) | 67(5) | 95(6) | 60(5) | -23(5) | -4(4) | 2(5) |
| C(24) | 65(5) | 69(5) | 56(5) | -33(4) | -6(4) | 21(4) |
| N(1) | 34(3) | 36(3) | 35(3) | -12(2) | -1(2) | -11(2) |
| N(2) | 36(3) | 40(3) | 35(3) | -10(2) | -2(2) | -5(2) |
| C(7X) | 45(4) | 75(5) | 61(5) | -29(4) | -11(3) | -1(4) |
| C(8X) | 37(3) | 48(4) | 48(4) | -11(3) | -2(3) | -5(3) |
| C(9X) | 44(4) | 44(4) | 60(4) | -23(3) | 1(3) | -9(3) |
| C(10X) | 48(4) | 64(5) | 51(4) | -20(4) | 8(3) | -5(3) |
| C(11X) | 38(4) | 58(4) | 47(4) | -16(4) | -1(3) | -5(3) |
| C(1X) | 43(4) | 58(5) | 105(6) | -41(5) | -7(4) | 0(3) |
| C(2X) | 39(3) | 60(4) | 48(4) | -27(3) | 6(3) | -16(3) |
| C(3X) | 45(4) | 55(4) | 64(5) | -26(4) | 12(3) | -22(3) |
| C(4X) | 42(3) | 41(4) | 41(4) | -10(3) | 8(3) | -11(3) |
| C(5X) | 45(4) | 47(4) | 48(4) | -3(3) | -5(3) | -19(3) |
| C(6X) | 58(5) | 44(4) | 103(6) | -6(4) | -14(4) | -8(3) |

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Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for ZnNDC.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(3A) | 2409 | 3853 | 9768 | 36 |
| H(5A) | 2332 | 5875 | 10096 | 43 |
| H(6A) | -1100 | 2493 | 9610 | 43 |
| H(9A) | 4836 | -2835 | 8722 | 52 |
| H(11B) | 5343 | -4675 | 8232 | 57 |
| H(12A) | 5661 | -6789 | 8582 | 53 |
| H(15A) | 2503 | 2958 | 5654 | 46 |
| H(16A) | 2856 | 4519 | 4327 | 50 |
| H(18A) | 4215 | 6241 | 3376 | 43 |
| H(20A) | 5172 | 9529 | 3702 | 102 |
| H(21A) | 3685 | 8070 | 4494 | 120 |
| H(22A) | 1527 | 8515 | 5242 | 104 |
| H(23A) | 1026 | 10638 | 5072 | 89 |
| H(24A) | 2536 | 12096 | 4336 | 74 |
| H(1N) | 490(50) | 920(50) | 8030(40) | 41 |
| H(7XA) | 3048 | 4554 | 7822 | 70 |
| H(7XB) | 2298 | 5862 | 7759 | 70 |
| H(7XC) | 3075 | 5595 | 6870 | 70 |
| H(8XA) | 1500 | 4181 | 6947 | 54 |
| H(8XB) | 723 | 4455 | 7832 | 54 |
| H(9XA) | -531 | 7437 | 6619 | 57 |
| H(9XB) | 114 | 6784 | 7555 | 57 |
| H(10A) | -2275 | 6787 | 7791 | 66 |
| H(10B) | -1641 | 5416 | 7996 | 66 |
| H(10C) | -2286 | 6068 | 7061 | 66 |
| H(11A) | 865 | 5292 | 5577 | 58 |
| H(1XA) | 2050 | -1759 | 7649 | 78 |
| H(1XB) | 847 | -1691 | 8416 | 78 |
| H(1XC) | 2139 | -911 | 8281 | 78 |
| H(2XA) | 247 | -527 | 6981 | 56 |

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| | | | | |
|---|---|---|---|---|
| H(2XB) | 1529 | 258 | 6857 | 56 |
| H(3XA) | -2845 | -812 | 8576 | 63 |
| H(3XB) | -1728 | -1081 | 7818 | 63 |
| H(3XC) | -2608 | 190 | 7614 | 63 |
| H(4XA) | -687 | -575 | 8923 | 50 |
| H(4XB) | -1571 | 691 | 8725 | 50 |
| H(5XA) | -1083 | 1457 | 6633 | 58 |
| H(5XB) | 253 | 2134 | 6609 | 58 |
| H(6XA) | -1687 | 3351 | 6868 | 85 |
| H(6XB) | -827 | 2986 | 7728 | 85 |
| H(6XC) | -2161 | 2302 | 7760 | 85 |

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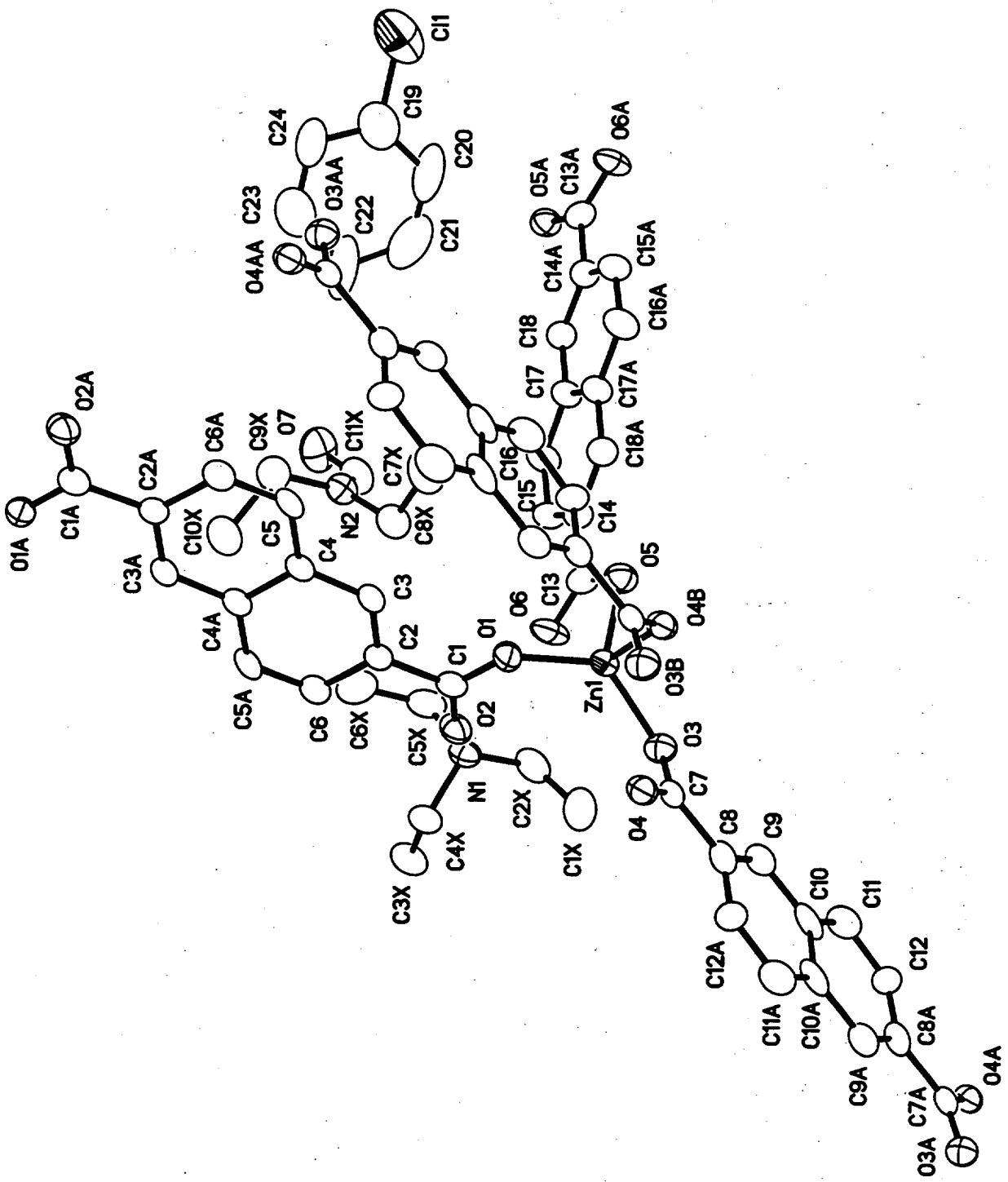

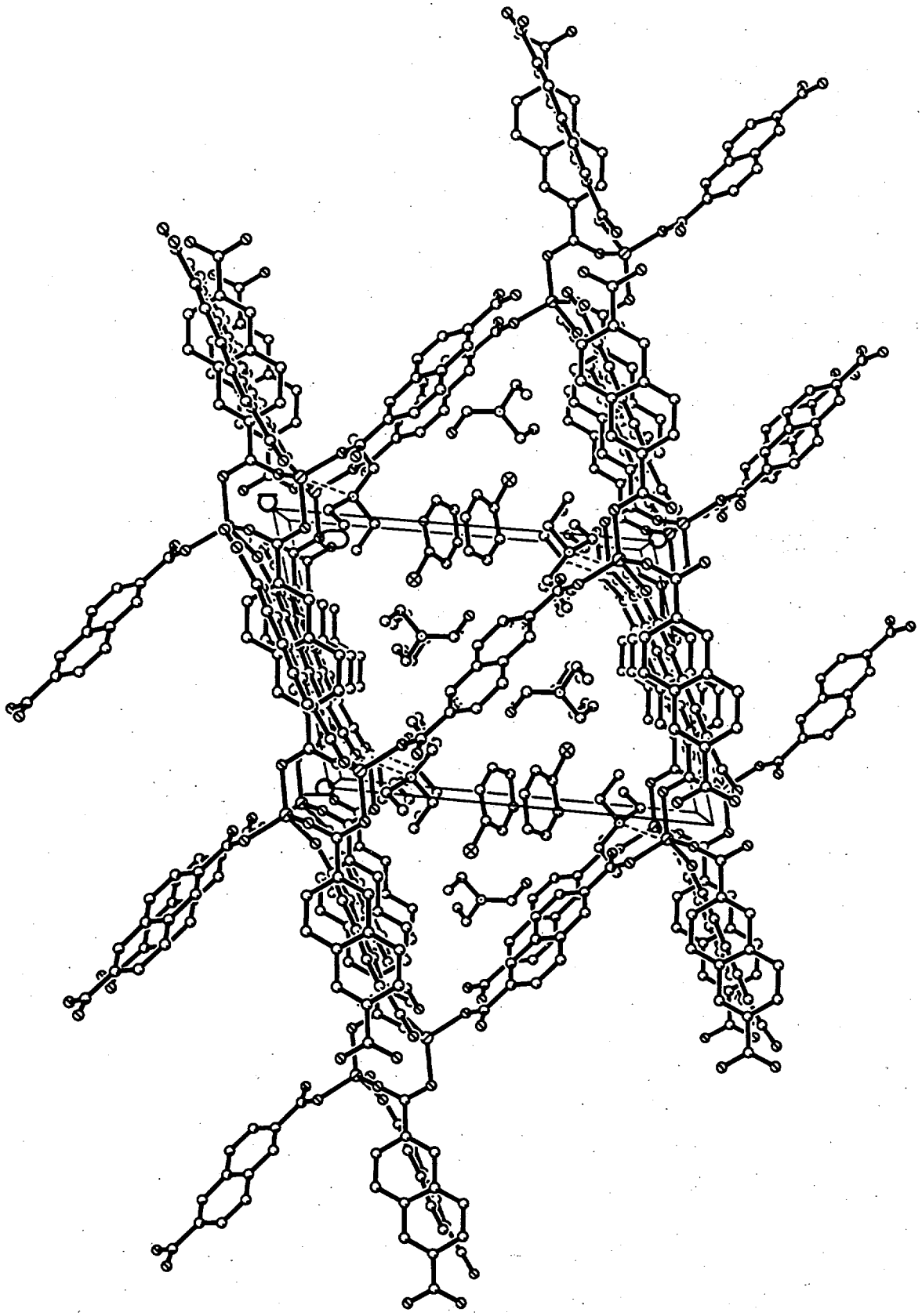

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*Introduction*

The structure analysis clearly shows that the zinc has formed clusters with a bridging oxygen betweeen three zinc ions. We presume that the oxygen bridge is an $O^{-2}$ oxo bridge. The empirical formula for the main framework is clearly $Zn_3O(C_9H_3O_6)2$. This framework extends in three dimensions to outline large channels parallel to the c-axis and smaller cavities between the channels.

As is usual for these structures the contents of the channels are highly disordered. In this case, since the empirical formula for the framework has two negative charges per Zn cluster, the contents must include cations – probably $[HNEt_3]^+$ based on the synthesis. The electron density in the cavities was modeled by partially occupied carbon atoms assigned fixed thermal parameters (8.5Å$^2$ for those atoms in the small cavity and 12.0Å$^2$ for those in the channels). The positions and occupancies of the atoms were allowed to refine. (An atom located in the center of the channel was assigned as a full-occupancy oxygen after refining to ridiculous values as a carbon.) Analysis of the occupancies show enough electron density in the small cavity to account for most of a triethylammonium cation per cavity (1/2 cation per zinc cluster) and enough in the large cavity to account for the rest of the cations needed for charge balance.

Each zinc cluster has an octahedral zinc and two tetrahedral zinc atoms. The octahedral zinc is coordinated by the carboxylate groups of several btc ligands the central oxo atom and by a DMF molecule which is only slightly disordered across a crystallographic mirror plane (in fact the C=O atoms are so close to the mirror plane that they are modelled with anisotropic thermal parameters rather than disorder). The tetrahedral zinc atoms are coordinated by the oxo atom and carboxylate oxygens from several btc ligands. In addition they are coordinated by an apparent oxygen atom (O9) which refines with a fixed Biso of 3.5Å$^2$ to an occupancy of 0.37. This might easily be assigned as a partially occupied water molecule if it were not for the fact that O9 is 1.7Å from another apparent oxygen atom, O10, which refines best as a full-occupancy oxygen atom. (Even changing it to a carbon atom significantly increases the R-value.) Due to the crystallographic mirror plane running through the position of O10, there are actually two sites for O9 which are 1.,7Å from O10. I have no idea what this actually is.

The btc anions are mostly planar, but the carboxylate moieties are twisted more than one might expect to see in an isolated molecule. Torsion angles in the Tables clearly show the magnitude of the twist. This is shown in the Figures attached.

A shelx output file is included on the diskette.

*Experimental*

Data Collection

A colorless blocklike crystal of $Zn_3O_{15.75}N_3C_{33}H_{44}$ having approximate dimensions of 0.05 x 0.18 x 0.21 mm was mounted on a glass fiber using Paratone N hydrocarbon oil. All measurements were made on a SMART[10] CCD area detector with graphite monochromated Mo-K$\alpha$ radiation.

Cell constants and an orientation matrix , obtained from a least-squares refinement using the measured positions of 8192 reflections in the range $3.00 < 2\theta < 45.00°$ corresponded to an I-centered tetragonal cell (laue class: 4/mmm) with dimensions:

a = 20.6571(7) $\AA$

c = 17.8400(6) $\AA$

V = 7612.6(4) $\AA^3$

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For Z = 8 and F.W. = 930.86, the calculated density is 1.62 g/cm$^3$. Based on the systematic absences of:

hkl: h+k+l $\neq$ 2n

0kl: l $\neq$ 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

I4cm (#108)

The data were collected at a temperature of -155 $\pm$ 1°C. Frames corresponding to an arbitrary hemisphere of data were collected using $\omega$ scans of 0.3° counted for a total of 10.0 seconds per frame.

Data Reduction

Data were integrated by the program SAINT[11] to a maximum $2\theta$ value of 49.4°. The data were corrected for Lorentz and polarization effects. Data were analyzed for agreement and possible absorption using XPREP[12]. An empirical absorption correction based on comparison of redundant and equivalent reflections as applied using XPREP (Tmax = 0.85, Tmin = 0.74).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. Carbon atoms and oxygen atoms in the ligand, and the "ordered" atoms of the DMF molecule were refined with anisotropic displacement parameters. All atoms in the void region were refined isotropically as described above. The correct enantiomorph of the crystal was determined by a comparison of the observed and calculated values of the structure factors of Friedel pairs. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 2500 observed reflections (I > 3.00$\sigma$(I)) and 263 variable parameters and converged (largest parameter shift was 0.08 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.042$$

$$R_w = \sqrt{(\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2)}] = 0.046$$

The standard deviation of an observation of unit weight[4] was 1.34. The weighting scheme was based on counting statistics and included a factor (p = 0.030) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.42 and -0.61 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta$f' and $\Delta$f" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

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*References*

(1) SIR92: Altomare, A., Cascarano, M., Giacovazzo, C., Guagliardi, A. (1993). J. Appl. Cryst., 26, 343.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

(10) SMART: Area-Detector Software Package, Bruker Analytical X-ray Systems, Inc.: Madison, WI, (1995-99)

(11) SAINT: SAX Area-Dectector Integration Program, V5.04; Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(12) XPREP:(v 5.03) Part of the SHELXTL Crystal Structure Determination Siemens Industrial Automation, Inc.: Madison, WI, (1995)

(13) SADABS: Siemens Area Detector ABSorption correction program, George Sheldrick, (1996). Advance copy, private communication.

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## *EXPERIMENTAL DETAILS*

### A. Crystal Data

| | |
|---|---|
| Empirical Formula | $Zn_3O_{15.75}N_3C_{33}H_{44}$ |
| Formula Weight | 930.86 |
| Crystal Color, Habit | colorless, blocklike |
| Crystal Dimensions | 0.05 X 0.18 X 0.21 mm |
| Crystal System | tetragonal |
| Lattice Type | I-centered |
| Lattice Parameters | a = 20.6571(7)Å<br>c = 17.8400(6) Å<br>V = 7612.6(4) $Å^3$ |
| Space Group | I4cm (#108) |
| Z value | 8 |
| $D_{calc}$ | 1.624 g/cm$^3$ |
| $F_{000}$ | 3832.00 |
| $\mu$(MoK$\alpha$) | 38.82 cm$^{-1}$ |

### B. Intensity Measurements

| | |
|---|---|
| Diffractometer | SMART CCD |
| Radiation | MoK$\alpha$ ($\lambda$ = 0.71069 Å)<br>graphite monochromated |
| Detector Position | 60.00 mm |
| Exposure Time | 10.0 seconds per frame. |
| Scan Type | $\omega$ (0.3 degrees per frame) |
| $2\theta_{max}$ | 49.4° |
| No. of Reflections Measured | Total: 17901 |

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| | |
|---|---|
| | Unique: 3082 ($R_{int}$ = 0.055) |
| Corrections | Lorentz-polarization<br>Absorption (Tmax = 0.85, Tmin = 0.74) |

C. Structure Solution and Refinement

| | |
|---|---|
| Structure Solution | Direct Methods (SIR92) |
| Refinement | Full-matrix least-squares |
| Function Minimized | $\Sigma w(\|Fo\| - \|Fc\|)^2$ |
| Least Squares Weights | $w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$ |
| p-factor | 0.0300 |
| Anomalous Dispersion | All non-hydrogen atoms |
| No. Observations (I>3.00$\sigma$(I)) | 2500 |
| No. Variables | 263 |
| Reflection/Parameter Ratio | 9.51 |
| Residuals: R; Rw; Rall | 0.042 ; 0.046; 0.064 |
| Goodness of Fit Indicator | 1.34 |
| Max Shift/Error in Final Cycle | 0.08 |
| Maximum peak in Final Diff. Map | 0.42 $e^-/\AA^3$ |
| Minimum peak in Final Diff. Map | -0.61 $e^-/\AA^3$ |

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Table 1. Atomic coordinates and $B_{iso}/B_{eq}$ and occupancy

| atom | x | y | z | $B_{eq}$ | occ |
|---|---|---|---|---|---|
| Zn(1) | 0.24525(4) | 0.37745(4) | 0.0000 | 1.905(15) | |
| Zn(2) | 0.15972(4) | 0.3403 | 0.14506(8) | 1.316(10) | 1/2 |
| O(1) | 0.2604(2) | 0.3364(3) | 0.1607(3) | 2.37(12) | |
| O(2) | 0.3113(2) | 0.3238(3) | 0.0514(3) | 2.63(12) | |
| O(3) | 0.5590(2) | 0.1671(2) | 0.1369(3) | 1.93(11) | |
| O(4) | 0.5393(3) | 0.2463(3) | 0.0529(3) | 2.61(12) | |
| O(5) | 0.4478(3) | 0.1890(3) | 0.3836(3) | 3.11(14) | |
| O(6) | 0.3713(3) | 0.2648(3) | 0.3935(3) | 3.04(13) | |
| O(7) | 0.1461(3) | 0.3539 | 0.2564(4) | 2.65(10) | 1/2 |
| O(8) | 0.1623(2) | 0.3377 | 0.0270(4) | 1.78(8) | 1/2 |
| O(9) | 0.1544(8) | 0.4138(7) | -0.0263(8) | 3.5000 | 0.37(2) |
| O(10) | 0.0739(3) | 0.4261 | -0.0357(5) | 4.19(12) | 1/2 |
| O(11) | 0.5000 | 0.5000 | 0.2282(6) | 4.30(15) | 1/4 |
| N(1) | 0.1757(7) | 0.3503 | 0.3765(8) | 4.5(4) | 1/2 |
| C(1) | 0.3625(3) | 0.2836(4) | 0.1586(4) | 1.89(16) | |
| C(2) | 0.4162(4) | 0.2646(4) | 0.1189(4) | 1.95(17) | |
| C(3) | 0.4668(3) | 0.2323(3) | 0.1536(4) | 2.05(16) | |
| C(4) | 0.4636(4) | 0.2188(4) | 0.2307(4) | 2.09(17) | |
| C(5) | 0.4105(4) | 0.2402(4) | 0.2717(4) | 1.88(16) | |
| C(6) | 0.3602(4) | 0.2723(4) | 0.2376(4) | 2.36(18) | |
| C(7) | 0.3061(4) | 0.3164(4) | 0.1216(4) | 2.25(18) | |
| C(8) | 0.5271(4) | 0.2118(4) | 0.1123(4) | 1.95(17) | |
| C(9) | 0.4111(4) | 0.2285(4) | 0.3560(4) | 2.32(18) | |
| C(10) | 0.1704(5) | 0.3296 | 0.3035(9) | 7.4(3) | 1/2 |
| C(11) | 0.1272(8) | 0.3970 | 0.4065(10) | 4.1(4) | 1/2 |
| C(12) | 0.2160(13) | 0.3166 | 0.4350(13) | 8.3(8) | 1/2 |
| C(13) | 0.0000 | 0.5000 | 0.2895(18) | 8.5000 | 0.26(2) |
| C(14) | -0.0439(15) | 0.5114(16) | 0.2297(17) | 8.5000 | 0.42(4) |
| C(15) | -0.0199(7) | 0.5199 | 0.1434(17) | 8.5000 | 0.41(3) |
| C(16) | 0.0000 | 0.5000 | 0.062(5) | 8.5000 | 0.09(2) |
| C(17) | 0.0750(7) | 0.5750 | 0.0907(13) | 8.5000 | 0.52(4) |
| C(18) | 0.0575(9) | 0.5575 | 0.1564(18) | 8.5000 | 0.39(3) |
| C(19) | 0.392(3) | 0.470(3) | 0.047(3) | 12.0000 | 0.37(6) |
| C(20) | 0.440(2) | 0.442(3) | 0.083(3) | 12.0000 | 0.46(7) |
| C(21) | 0.4359(13) | 0.494(2) | 0.1211(18) | 12.0000 | 0.67(7) |
| C(22) | 0.407(3) | 0.444(3) | 0.149(4) | 12.0000 | 0.37(6) |

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Table 1. Atomic coordinates and $B_{iso}/B_{eq}$ and occupancy (continued)

| atom | x | y | z | $B_{eq}$ | occ |
|---|---|---|---|---|---|
| C(23) | 0.398(3) | 0.485(3) | 0.227(3) | 12.0000 | 0.40(8) |
| C(24) | 0.4109(18) | 0.506(2) | 0.321(2) | 12.0000 | 0.52(5) |
| C(25) | 0.369(2) | 0.460(2) | 0.274(3) | 12.0000 | 0.61(8) |
| C(26) | 0.345(2) | 0.459(2) | 0.341(3) | 12.0000 | 0.65(10) |
| C(27) | 0.377(2) | 0.495(3) | 0.420(2) | 12.0000 | 0.46(6) |
| C(28) | 0.395(3) | 0.440(3) | 0.468(4) | 12.0000 | 0.32(6) |
| C(29) | 0.3420(16) | 0.4261(18) | 0.380(2) | 12.0000 | 0.77(11) |
| C(30) | 0.285(2) | 0.4154(16) | 0.363(2) | 12.0000 | 0.62(6) |
| C(31) | 0.436(4) | 0.462(4) | 0.413(4) | 12.0000 | 0.28(6) |
| C(32) | 0.423(4) | 0.562(5) | 0.280(5) | 12.0000 | 0.24(6) |
| H(1) | 0.4184 | 0.2744 | 0.0668 | 2.4264 | |
| H(2) | 0.4965 | 0.1933 | 0.2545 | 2.4954 | |
| H(3) | 0.3239 | 0.2870 | 0.2661 | 2.8705 | |
| H(4) | 0.2011 | 0.2989 | 0.2948 | 7.3787 | 1/2 |

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

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Table 2. Anisotropic Displacement Parameters

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| Zn(1) | 0.0282(4) | 0.0298(4) | 0.0143(3) | 0.0034(4) | 0.0014(5) | -0.0010(4) |
| Zn(2) | 0.0204(4) | 0.0204 | 0.0092(5) | 0.0038(5) | 0.0006 | -0.0006(4) |
| O(1) | 0.016(3) | 0.055(4) | 0.019(3) | 0.016(3) | 0.002(2) | 0.003(3) |
| O(2) | 0.032(3) | 0.054(4) | 0.014(3) | 0.022(3) | -0.003(2) | -0.004(3) |
| O(3) | 0.025(3) | 0.027(3) | 0.021(3) | 0.004(2) | 0.001(3) | 0.006(3) |
| O(4) | 0.031(3) | 0.045(4) | 0.023(3) | 0.008(3) | 0.008(2) | 0.008(3) |
| O(5) | 0.037(4) | 0.060(4) | 0.021(3) | 0.017(3) | 0.001(3) | 0.015(3) |
| O(6) | 0.046(4) | 0.054(4) | 0.015(3) | 0.009(3) | 0.001(3) | 0.006(3) |
| O(7) | 0.041(3) | 0.0410 | 0.019(4) | 0.005(4) | -0.0044 | 0.004(3) |
| O(8) | 0.028(3) | 0.0285 | 0.011(3) | -0.003(3) | 0.0012 | -0.001(2) |
| O(10) | 0.060(4) | 0.0603 | 0.039(5) | 0.015(5) | -0.0082 | 0.008(3) |
| O(11) | 0.074(6) | 0.0740 | 0.015(6) | 0.0000 | 0.0000 | 0.0000 |
| C(1) | 0.023(4) | 0.035(4) | 0.014(4) | 0.001(3) | -0.004(3) | -0.001(3) |
| C(2) | 0.027(5) | 0.037(5) | 0.010(3) | 0.000(4) | -0.002(3) | 0.003(3) |
| C(3) | 0.021(4) | 0.038(4) | 0.018(4) | 0.004(3) | 0.002(3) | 0.006(4) |
| C(4) | 0.026(4) | 0.028(4) | 0.025(4) | 0.008(4) | 0.001(4) | 0.003(4) |
| C(5) | 0.024(4) | 0.039(5) | 0.008(3) | 0.002(3) | 0.002(3) | 0.009(3) |
| C(6) | 0.030(5) | 0.041(5) | 0.018(4) | 0.001(4) | 0.005(3) | 0.003(4) |
| C(7) | 0.032(5) | 0.040(5) | 0.013(4) | 0.001(4) | -0.009(3) | 0.005(3) |
| C(8) | 0.023(4) | 0.032(5) | 0.020(4) | -0.001(4) | 0.005(3) | -0.003(3) |
| C(9) | 0.030(5) | 0.044(5) | 0.014(4) | -0.001(4) | 0.004(4) | 0.004(4) |
| C(10) | 0.119(11) | 0.1187 | 0.042(9) | 0.083(13) | 0.0122 | -0.012(6) |

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

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Table 3. Bond Lengths(Å)

| atom | atom | distance |
|---|---|---|
| ZN1 | O2 | 1.983(5) |
| ZN1 | O6 | 1.946(5) |
| ZN1 | O9 | 2.076(15) |
| ZN2 | O1 | 2.100(5) |
| ZN2 | O3 | 2.092(5) |
| ZN2 | O8 | 2.108(6) |
| O2 | C7 | 1.266(8) |
| O4 | C8 | 1.302(8) |
| O6 | C9 | 1.297(9) |
| O9 | O10 | 1.690(16) |
| N1 | C10 | 1.375(18) |
| N1 | C11 | 1.661(14) |
| N1 | C12 | 1.73(3) |
| C1 | C6 | 1.430(9) |
| C2 | C3 | 1.386(10) |
| C3 | C8 | 1.509(10) |
| C5 | C6 | 1.375(10) |
| C11 | C11 | 0.71(2) |
| C13 | C14 | 1.42(3) |
| C13 | C14 | 1.42(3) |
| C14 | C14 | 0.95(7) |
| C14 | C15 | 1.63(4) |
| C15 | C16 | 1.56(8) |
| C15 | C18 | 1.79(3) |
| C19 | C20 | 1.32(6) |
| C20 | C21 | 1.26(5) |
| C20 | C22 | 1.37(7) |
| C22 | C23 | 1.65(8) |
| C23 | C25 | 1.15(6) |
| C24 | C25 | 1.54(5) |
| C24 | C32 | 1.39(9) |
| C25 | C32 | 1.61(10) |
| C26 | C29 | 0.97(4) |
| C27 | C28 | 1.47(7) |
| C27 | C31 | 1.40(8) |
| C29 | C30 | 1.23(4) |

| atom | atom | distance |
|---|---|---|
| ZN1 | O4 | 1.961(5) |
| ZN1 | O8 | 1.960(3) |
| ZN2 | O1 | 2.100(5) |
| ZN2 | O3 | 2.092(5) |
| ZN2 | O7 | 2.025(7) |
| O1 | C7 | 1.244(9) |
| O3 | C8 | 1.215(9) |
| O5 | C9 | 1.218(9) |
| O7 | C10 | 1.102(17) |
| N1 | N1 | 0.76(2) |
| N1 | C11 | 1.490(16) |
| N1 | C12 | 1.51(2) |
| C1 | C2 | 1.373(10) |
| C1 | C7 | 1.500(10) |
| C3 | C4 | 1.405(10) |
| C4 | C5 | 1.391(10) |
| C5 | C9 | 1.525(9) |
| C12 | C12 | 0.95(4) |
| C13 | C14 | 1.42(3) |
| C13 | C14 | 1.42(3) |
| C14 | C14 | 1.62(7) |
| C15 | C15 | 1.16(4) |
| C15 | C18 | 1.79(3) |
| C17 | C18 | 1.28(3) |
| C19 | C21 | 1.67(6) |
| C20 | C21 | 1.53(6) |
| C21 | C22 | 1.30(6) |
| C23 | C24 | 1.75(7) |
| C23 | C32 | 1.78(11) |
| C24 | C26 | 1.72(6) |
| C25 | C26 | 1.29(5) |
| C26 | C27 | 1.73(5) |
| C26 | C30 | 1.57(6) |
| C27 | C29 | 1.75(5) |
| C28 | C31 | 1.37(7) |

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Table 4. Bond Lengths($\AA$)

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C2 | H1 | 0.95 | C4 | H2 | 0.96 |
| C6 | H3 | 0.96 | C10 | H4 | 0.91 |

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Table 5. Bond Angles(°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| O2 | ZN1 | O4 | 105.0(2) | O2 | ZN1 | O6 | 105.8(2) |
| O2 | ZN1 | O8 | 104.7(3) | O2 | ZN1 | O9 | 158.3(5) |
| O4 | ZN1 | O6 | 121.6(2) | O4 | ZN1 | O8 | 105.0(2) |
| O4 | ZN1 | O9 | 78.6(5) | O6 | ZN1 | O8 | 113.2(3) |
| O6 | ZN1 | O9 | 89.4(4) | O8 | ZN1 | O9 | 54.3(4) |
| O1 | ZN2 | O1 | 84.7(3) | O1 | ZN2 | O3 | 172.7(2) |
| O1 | ZN2 | O3 | 88.5(2) | O1 | ZN2 | O7 | 90.8(2) |
| O1 | ZN2 | O8 | 96.1(2) | O1 | ZN2 | O3 | 88.5(2) |
| O1 | ZN2 | O3 | 172.7(2) | O1 | ZN2 | O7 | 90.8(2) |
| O1 | ZN2 | O8 | 96.1(2) | O3 | ZN2 | O3 | 98.1(3) |
| O3 | ZN2 | O7 | 86.6(2) | O3 | ZN2 | O8 | 87.4(2) |
| O3 | ZN2 | O7 | 86.6(2) | O3 | ZN2 | O8 | 87.4(2) |
| O7 | ZN2 | O8 | 170.7(3) | ZN2 | O1 | C7 | 133.6(5) |
| ZN1 | O2 | C7 | 117.8(5) | ZN2 | O3 | C8 | 128.3(5) |
| ZN1 | O4 | C8 | 123.7(5) | ZN1 | O6 | C9 | 110.0(5) |
| ZN2 | O7 | C10 | 128.5(10) | ZN1 | O8 | ZN1 | 132.3(4) |
| ZN1 | O8 | ZN2 | 104.9(2) | ZN1 | O8 | ZN2 | 104.9(2) |
| ZN1 | O9 | O10 | 165.1(9) | O9 | O10 | O9 | 72.2(11) |
| C10 | N1 | C11 | 119.2(13) | C10 | N1 | C12 | 123.7(11) |
| C11 | N1 | C12 | 115.0(13) | C2 | C1 | C6 | 119.2(6) |
| C2 | C1 | C7 | 122.0(6) | C6 | C1 | C7 | 118.7(6) |
| C1 | C2 | C3 | 121.1(6) | C2 | C3 | C4 | 119.9(6) |
| C2 | C3 | C8 | 122.6(7) | C4 | C3 | C8 | 117.5(6) |
| C3 | C4 | C5 | 119.2(7) | C4 | C5 | C6 | 121.2(6) |
| C4 | C5 | C9 | 117.6(6) | C6 | C5 | C9 | 121.2(6) |
| C1 | C6 | C5 | 119.3(6) | O1 | C7 | O2 | 125.4(7) |
| O1 | C7 | C1 | 119.5(6) | O2 | C7 | C1 | 115.0(7) |
| O3 | C8 | O4 | 127.3(7) | O3 | C8 | C3 | 118.9(6) |
| O4 | C8 | C3 | 113.8(7) | O5 | C9 | O6 | 125.0(7) |
| O5 | C9 | C5 | 120.6(7) | O6 | C9 | C5 | 114.4(7) |
| O7 | C10 | N1 | 128.1(13) | | | | |

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Table 6. Bond Angles(°)

| atom | atom | atom | angle |
|---|---|---|---|
| C1 | C2 | H1 | 118.7 |
| C3 | C4 | H2 | 120.7 |
| C1 | C6 | H3 | 119.9 |
| O7 | C10 | H4 | 120.3 |
| N1 | C10 | H4 | 108.9 |
| C3 | C2 | H1 | 120.2 |
| C5 | C4 | H2 | 120.0 |
| C5 | C6 | H3 | 120.8 |
| N1 | C10 | H4 | 108.9 |

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Table 7. Torsion Angles(°)

| atom | atom | atom | atom | angle | atom | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|---|---|
| ZN1 | O2 | C7 | O1 | 5.1(11) | ZN1 | O2 | C7 | C1 | -171.5(5) |
| ZN1 | O8 | ZN1 | O2 | 68.9(5) | ZN1 | O8 | ZN1 | O6 | -45.8(5) |
| ZN1 | O8 | ZN1 | O9 | -117.1(7) | ZN1 | O8 | ZN2 | O1 | -28.5(3) |
| ZN1 | O8 | ZN2 | O1 | -113.8(3) | ZN1 | O8 | ZN2 | O3 | 158.0(3) |
| ZN1 | O8 | ZN2 | O3 | 59.8(3) | ZN1 | O8 | ZN2 | O7 | 108.9(3) |
| ZN1 | O9 | O10 | O9 | -26.5(39) | ZN2 | O1 | C7 | O2 | 40.4(12) |
| ZN2 | O1 | C7 | C1 | -143.3(6) | ZN2 | O1 | C7 | O2 | -40.4(12) |
| ZN2 | O1 | C7 | C1 | 143.3(6) | ZN2 | O7 | C10 | N1 | 159.4(8) |
| ZN2 | O7 | C10 | N1 | -159.4(8) | ZN2 | O8 | ZN1 | O2 | 58.0(3) |
| ZN2 | O8 | ZN1 | O4 | -52.4(3) | ZN2 | O8 | ZN1 | O6 | 172.7(2) |
| ZN2 | O8 | ZN1 | O9 | -116.0(6) | ZN2 | O8 | ZN1 | O2 | -58.0(3) |
| ZN2 | O8 | ZN1 | O6 | -172.7(2) | ZN2 | O8 | ZN1 | O9 | 116.0(6) |
| O1 | ZN2 | O1 | C7 | -72.2(7) | O1 | ZN2 | O7 | C10 | -42.34(15) |
| O1 | C7 | C1 | C2 | -175.4(7) | O1 | C7 | C1 | C6 | 4.3(11) |
| O2 | ZN1 | O9 | O10 | -16.0(45) | O2 | C7 | C1 | C2 | 1.4(11) |
| O2 | C7 | C1 | C6 | -179.0(7) | O3 | ZN2 | O3 | C8 | 151.4(5) |
| O3 | C8 | C3 | C2 | -156.2(7) | O3 | C8 | C3 | C4 | 25.1(10) |
| O4 | C8 | C3 | C2 | 24.8(10) | O4 | C8 | C3 | C4 | -153.8(7) |
| O5 | C9 | C5 | C4 | -17.4(12) | O5 | C9 | C5 | C6 | 164.5(8) |
| O6 | C9 | C5 | C4 | 160.9(7) | O6 | C9 | C5 | C6 | -17.2(11) |
| O7 | ZN2 | O1 | C7 | 162.9(7) | O7 | ZN2 | O1 | C7 | -162.9(7) |
| O7 | C10 | N1 | C11 | 23.7(12) | O7 | C10 | N1 | C12 | -173.4(12) |
| O8 | ZN1 | O2 | C7 | -55.4(6) | O8 | ZN1 | O9 | O10 | 0.0(33) |
| O8 | ZN1 | O2 | C7 | 55.4(6) | O8 | ZN1 | O9 | O10 | 0.0(33) |
| O8 | ZN2 | O1 | C7 | -23.4(7) | O8 | ZN2 | O1 | C7 | 23.4(7) |
| O8 | ZN2 | O7 | C10 | 180.00000(10) | O9 | ZN1 | O2 | C7 | -41.9(14) |
| C1 | C2 | C3 | C4 | -0.2(12) | C1 | C2 | C3 | C8 | -178.8(7) |
| C1 | C6 | C5 | C4 | 0.4(13) | C1 | C6 | C5 | C9 | 178.4(7) |
| C2 | C1 | C6 | C5 | -2.9(12) | C2 | C3 | C4 | C5 | -2.3(12) |
| C3 | C2 | C1 | C6 | 2.8(12) | C3 | C2 | C1 | C7 | -177.6(7) |
| C3 | C4 | C5 | C6 | 2.2(12) | C3 | C4 | C5 | C9 | -175.9(7) |
| C5 | C4 | C3 | C8 | 176.4(7) | C5 | C6 | C1 | C7 | 177.5(7) |

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Table 8. Non-bonded Contacts out to 3.83 Å

| atom | atom | distance | ADC |
|---|---|---|---|
| ZN1 | C19 | 3.69(6) | 1 |
| O2 | C28 | 3.38(7) | 55415 |
| O2 | C22 | 3.61(6) | 1 |
| O2 | C20 | 3.67(5) | 1 |
| O3 | O8 | 2.901(7) | 4 |
| O3 | C15 | 3.148(18) | 54503 |
| O3 | C14 | 3.19(3) | 6 |
| O3 | C17 | 3.458(8) | 54503 |
| O3 | O10 | 3.645(9) | 4 |
| O4 | O9 | 2.558(17) | 65505 |
| O4 | O8 | 3.111(5) | 4 |
| O4 | O6 | 3.411(7) | 65411 |
| O4 | C9 | 3.676(9) | 65411 |
| O4 | C22 | 3.74(5) | 65505 |
| O5 | O10 | 2.816(9) | 10 |
| O5 | O8 | 3.467(8) | 10 |
| O5 | C11 | 3.475(9) | 65505 |
| O6 | O8 | 3.260(7) | 10 |
| O6 | C30 | 3.63(3) | 1 |
| O7 | C14 | 3.52(3) | 56502 |
| O7 | C30 | 3.67(4) | 1 |
| O8 | C9 | 3.670(9) | 55410 |
| O10 | C15 | 3.56(3) | 56502 |
| O10 | C13 | 3.79(3) | 55411 |
| O10 | C17 | 3.81(2) | 56502 |
| O11 | C23 | 2.13(6) | 66502 |
| O11 | C23 | 2.13(6) | 56506 |
| O11 | C32 | 2.24(8) | 66502 |
| O11 | C32 | 2.24(8) | 56506 |
| O11 | C21 | 2.33(3) | 66502 |
| O11 | C21 | 2.33(3) | 56506 |
| O11 | C24 | 2.48(4) | 66502 |
| O11 | C24 | 2.48(4) | 56506 |
| O11 | C22 | 2.66(6) | 66502 |
| O11 | C22 | 2.66(6) | 56506 |
| O11 | C25 | 2.95(5) | 66502 |

| atom | atom | distance | ADC |
|---|---|---|---|
| O1 | C22 | 3.75(6) | 1 |
| O2 | C19 | 3.46(6) | 1 |
| O2 | C12 | 3.611(15) | 55410 |
| O3 | O7 | 2.823(8) | 4 |
| O3 | O9 | 2.977(15) | 65505 |
| O3 | O3 | 3.160(9) | 54507 |
| O3 | C18 | 3.322(6) | 54503 |
| O3 | C14 | 3.63(3) | 54503 |
| O3 | C10 | 3.761(16) | 4 |
| O4 | C19 | 3.02(6) | 65505 |
| O4 | O5 | 3.254(8) | 65411 |
| O4 | C7 | 3.451(9) | 65505 |
| O4 | C27 | 3.71(5) | 55415 |
| O5 | O9 | 1.894(15) | 15 |
| O5 | O9 | 3.398(17) | 10 |
| O5 | C30 | 3.47(3) | 65505 |
| O6 | O9 | 2.832(16) | 15 |
| O6 | C29 | 3.40(4) | 1 |
| O6 | C27 | 3.64(5) | 65505 |
| O7 | C14 | 3.52(3) | 45507 |
| O7 | C30 | 3.67(4) | 8 |
| O10 | C16 | 2.78(6) | 1 |
| O10 | C9 | 3.746(10) | 55410 |
| O10 | C17 | 3.81(2) | 1 |
| O11 | C23 | 2.13(6) | 1 |
| O11 | C23 | 2.13(6) | 65505 |
| O11 | C32 | 2.24(8) | 1 |
| O11 | C32 | 2.24(8) | 65505 |
| O11 | C21 | 2.33(3) | 1 |
| O11 | C21 | 2.33(3) | 65505 |
| O11 | C24 | 2.48(4) | 1 |
| O11 | C24 | 2.48(4) | 65505 |
| O11 | C22 | 2.66(6) | 1 |
| O11 | C22 | 2.66(6) | 65505 |
| O11 | C25 | 2.95(5) | 1 |
| O11 | C25 | 2.95(5) | 65505 |

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Table 8. Non-bonded Contacts out to 3.83 Å (continued)

| atom | atom | distance | ADC |
|---|---|---|---|
| O11 | C25 | 2.95(5) | 56506 |
| O11 | C20 | 3.10(5) | 66502 |
| O11 | C20 | 3.10(5) | 56506 |
| O11 | C31 | 3.63(7) | 66502 |
| O11 | C31 | 3.63(7) | 56506 |
| N1 | C30 | 3.38(4) | 8 |
| C1 | C22 | 3.43(6) | 1 |
| C2 | C19 | 3.75(6) | 65505 |
| C3 | C17 | 3.546(14) | 54503 |
| C3 | C23 | 3.80(5) | 65505 |
| C4 | C18 | 3.61(2) | 54503 |
| C4 | C30 | 3.70(3) | 65505 |
| C5 | C26 | 3.68(5) | 65505 |
| C5 | C32 | 3.83(9) | 65505 |
| C7 | C22 | 3.38(6) | 1 |
| C8 | C18 | 3.719(13) | 54503 |
| C9 | C30 | 3.77(3) | 65505 |
| C10 | C30 | 3.14(4) | 1 |
| C11 | C30 | 3.38(4) | 1 |
| C12 | C30 | 2.80(4) | 1 |
| C12 | C30 | 3.67(4) | 8 |
| C19 | C28 | 2.33(8) | 55415 |
| C19 | C27 | 2.40(7) | 56412 |
| C19 | C31 | 2.92(9) | 56412 |
| C19 | C19 | 3.27(8) | 56506 |
| C19 | C27 | 3.66(7) | 55415 |
| C20 | C28 | 2.28(8) | 55415 |
| C20 | C21 | 2.82(4) | 56506 |
| C20 | C31 | 3.08(9) | 55415 |
| C20 | C22 | 3.33(8) | 56506 |
| C20 | C20 | 3.42(9) | 66502 |
| C20 | C31 | 3.62(8) | 56412 |
| C21 | C22 | 2.13(8) | 56506 |
| C21 | C23 | 3.11(7) | 56506 |
| C21 | C28 | 3.41(7) | 55415 |
| C21 | C27 | 3.80(5) | 56412 |
| O11 | C20 | 3.10(5) | 1 |
| O11 | C20 | 3.10(5) | 65505 |
| O11 | C31 | 3.63(7) | 1 |
| O11 | C31 | 3.63(7) | 65505 |
| N1 | C30 | 2.64(4) | 1 |
| N1 | C29 | 3.78(4) | 1 |
| C2 | C22 | 3.74(6) | 1 |
| C2 | C20 | 3.76(5) | 1 |
| C3 | C18 | 3.647(17) | 54503 |
| C4 | C25 | 3.57(4) | 65505 |
| C4 | C26 | 3.64(5) | 65505 |
| C4 | C14 | 3.74(3) | 6 |
| C5 | C25 | 3.78(4) | 65505 |
| C6 | C32 | 3.59(8) | 65505 |
| C8 | C17 | 3.547(8) | 54503 |
| C9 | C26 | 3.62(4) | 65505 |
| C9 | C27 | 3.80(5) | 65505 |
| C10 | C30 | 3.14(4) | 8 |
| C11 | C17 | 3.51(3) | 56512 |
| C12 | C29 | 3.59(4) | 1 |
| C19 | C20 | 2.21(8) | 56506 |
| C19 | C28 | 2.34(8) | 56412 |
| C19 | C31 | 2.89(9) | 55415 |
| C19 | C21 | 3.14(7) | 56506 |
| C19 | C22 | 3.30(8) | 56506 |
| C19 | C29 | 3.82(7) | 56412 |
| C20 | C20 | 2.42(6) | 56506 |
| C20 | C21 | 2.95(5) | 66502 |
| C20 | C28 | 3.32(8) | 56412 |
| C20 | C27 | 3.40(7) | 55415 |
| C20 | C27 | 3.44(7) | 56412 |
| C21 | C21 | 1.88(4) | 56506 |
| C21 | C21 | 2.66(5) | 66502 |
| C21 | C28 | 3.18(7) | 56412 |
| C21 | C22 | 3.54(7) | 66502 |
| C21 | C31 | 3.83(8) | 56412 |

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Table 8. Non-bonded Contacts out to 3.83 Å (continued)

| atom | atom | distance | ADC |
|---|---|---|---|
| C22 | C22 | 3.19(8) | 56506 |
| C23 | C23 | 3.02(8) | 56506 |
| C23 | C24 | 3.53(6) | 56506 |
| C24 | C24 | 2.61(5) | 56506 |
| C24 | C26 | 3.25(6) | 56506 |
| C24 | C28 | 3.38(7) | 56506 |
| C24 | C32 | 3.52(8) | 56506 |
| C24 | C24 | 3.69(7) | 66502 |
| C25 | C31 | 3.80(9) | 56506 |
| C27 | C31 | 2.26(10) | 56506 |
| C27 | C29 | 3.59(6) | 56506 |
| C28 | C31 | 3.07(10) | 56506 |
| C29 | C31 | 3.82(9) | 56506 |
| C31 | C31 | 3.07(14) | 66502 |
| C32 | C32 | 2.88(11) | 56506 |
| C22 | C28 | 3.46(9) | 55415 |
| C23 | C25 | 3.39(8) | 56506 |
| C24 | C31 | 2.29(8) | 56506 |
| C24 | C25 | 2.89(7) | 56506 |
| C24 | C29 | 3.32(6) | 56506 |
| C24 | C27 | 3.46(7) | 56506 |
| C24 | C31 | 3.62(8) | 66502 |
| C24 | C32 | 3.78(8) | 66502 |
| C26 | C31 | 3.50(9) | 56506 |
| C27 | C28 | 2.75(8) | 56506 |
| C27 | C27 | 3.59(6) | 56506 |
| C28 | C28 | 3.53(9) | 56506 |
| C31 | C31 | 2.17(10) | 56506 |
| C31 | C32 | 3.78(11) | 66502 |

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The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ±4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| (1) | X, | Y, | Z | (2) | -X, | -Y, | Z |
| (3) | 1/2-X, | 1/2+Y, | Z | (4) | 1/2+X, | 1/2-Y, | Z |
| (5) | -Y, | X, | Z | (6) | Y, | -X, | Z |
| (7) | 1/2+Y, | 1/2+X, | Z | (8) | 1/2-Y, | 1/2-X, | Z |

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Table 9. Least Squares Planes

Plane number 1

| Atoms defining plane | Distance |
|---|---|
| C1 | -0.018(7) |
| C2 | 0.009(8) |
| C3 | 0.007(7) |
| C4 | -0.018(8) |
| C5 | 0.008(8) |
| C6 | 0.013(8) |

| Additional Atoms | Distance |
|---|---|
| C7 | -0.070 |
| C8 | 0.062 |
| C9 | 0.076 |
| O1 | -0.005 |
| O2 | -0.114 |
| O3 | -0.369 |
| O4 | 0.608 |
| O5 | -0.208 |
| O6 | 0.482 |
| ZN1 | 0.076 |
| ZN2 | -0.921 |

Summary

| plane | mean deviation | $\chi^2$ |
|---|---|---|
| 1 | 0.0120 | 17.8 |

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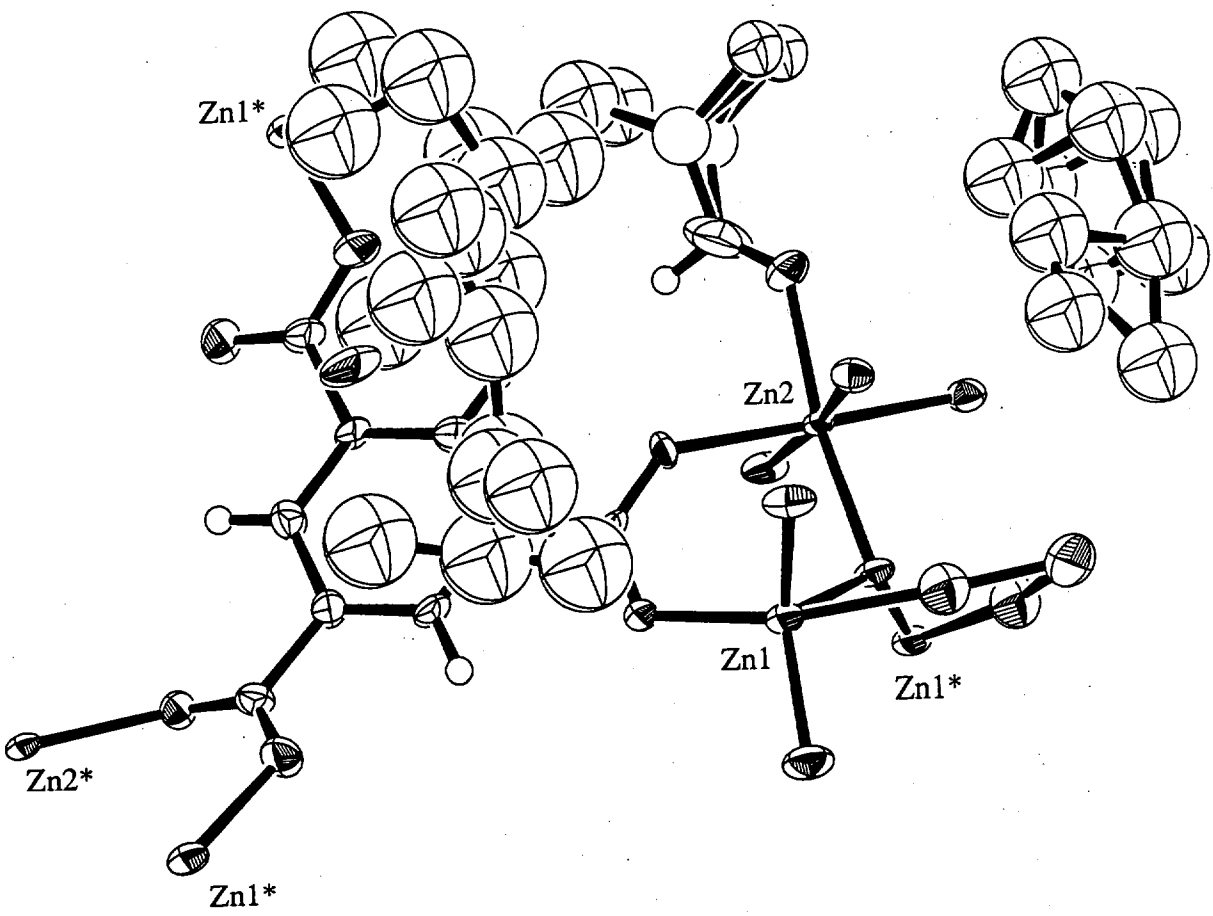

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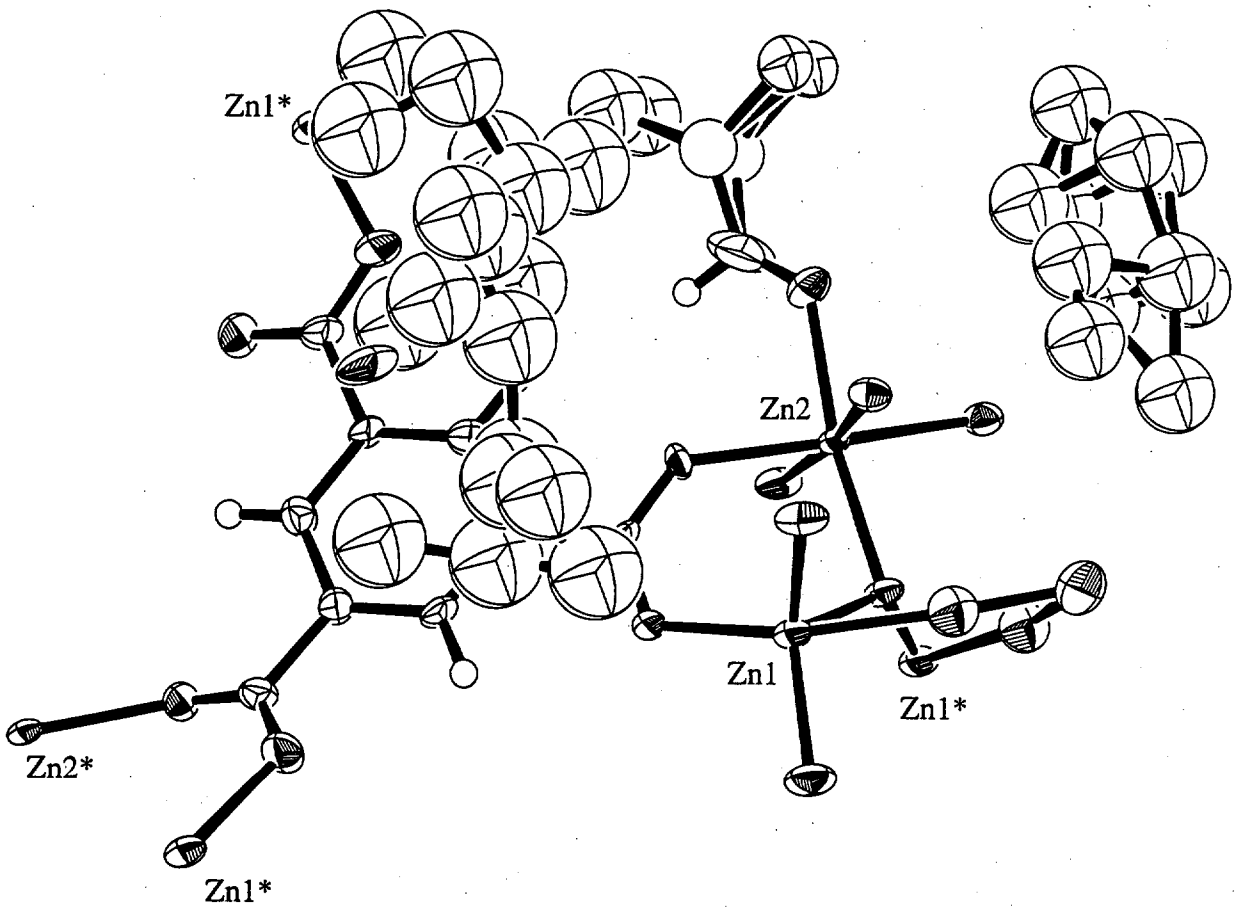

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**Structure Determination.**

Pale yellow plates of **zn90** were crystallized from a mixture of DMF/ethanol/water at room temperature.. A crystal of dimensions 0.40 x 0.32 x 0.06 mm was mounted on a standard Bruker SMART CCD-based X-ray diffractometer equipped with a normal focus Mo-target X-ray tube ($\lambda$ = 0.71073 A) operated at 2000 W power (50 kV, 40 mA). The X-ray intensities were measured at 158(2) K; the detector was placed at a distance of 5.508 cm from the crystal. A total of 2332 frames were collected with a scan width of 0.3° in $\omega$ and phi with an exposure time of 60 s/frame. The frames were integrated with the Bruker SAINT software package with a narrow frame algorithm. The integration of the data yielded a total of 79244 reflections to a maximum $2\theta$ value of 49.5° of which 8750 were independent and 4743 were greater that $2\sigma(I)$. The final cell constants (Table 1) were based on the xyz centroids of 5692 reflections above $10\sigma(I)$. Analysis of the data showed negligible decay during data collection; the data were processed with SADABS and corrected for absorption. The structure was solved and refined with the Bruker SHELXTL (version 5.10) software package, using the space group Pnma with Z = 4 for the formula $C_{55.5}H_{44}N_{0.5}O_{17}Zn_3$ which includes the contribution of partially occupied DMF and water lattice solvates. Use of the SQUEEZE routine of the PLATON program suite suggested the presence of significant additional solvent present in two large solvent accessible voids. Due to the uncertain composition of these solvates, their contribution to the derived crystal quantities has not been included. All non-hydrogen atoms were refined anisotropically with the hydrogen atoms located on a difference Fourier map and allowed to refine isotropically or placed in idealized positions. Full matrix least-squares refinement based on $F^2$ converged at R1 = 0.0797 and wR2 = 0.2246 [based on I > 2sigma(I)], R1 = 0.1120 and wR2 = 0.2414 for all data. Additional details are presented in Table 1 and are given as Supporting information as a CIF file.

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Sheldrick, G.M. SADABS. Program for Empirical Absorption Correction of Area Detector Data, University of Gottingen: Gottingen, Germany, 1996.
Saint Plus, v. 6.02, Bruker Analytical X-ray, Madison, WI, 1999.
PLATON. Spek, A.L. (1990) Acta Cryst. A46, C-34.

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Table 1. Crystal data and structure refinement for zn90.

| | |
|---|---|
| Identification code | zn90 |
| Empirical formula | C55.50 H44 N0.50 O17 Zn3 |
| Formula weight | 1186.02 |
| Temperature | 158(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Orthorhombic, Pnma |
| Unit cell dimensions | a = 17.158(6) A alpha = 90 deg.<br>b = 21.591(7) A beta = 90 deg.<br>c = 25.308(8) A gamma = 90 deg. |
| Volume | 9376(5) A^3 |
| Z, Calculated density | 4, 0.840 Mg/m^3 |
| Absorption coefficient | 0.802 mm^-1 |
| F(000) | 2426 |
| Crystal size | 0.06 x 0.32 x 0.40 mm |
| Theta range for data collection | 1.24 to 24.74 deg. |
| Limiting indices | -20<=h<=20, -25<=k<=25, -29<=l<=29 |
| Reflections collected / unique | 79244 / 8232 [R(int) = 0.0509] |
| Completeness to theta = 24.74 | 99.8 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.901 and 0.403 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 8232 / 14 / 400 |
| Goodness-of-fit on F^2 | 0.947 |
| Final R indices [I>2sigma(I)] | R1 = 0.0797, wR2 = 0.2226 |
| R indices (all data) | R1 = 0.1120, wR2 = 0.2414 |
| Extinction coefficient | 0.00020(11) |
| Largest diff. peak and hole | 0.991 and -0.533 e.A^-3 |

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Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for zn90.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Zn(1) | 5050(1) | 3350(1) | 3869(1) | 44(1) |
| Zn(2) | 6168(1) | 2500 | 3171(1) | 35(1) |
| O(1) | 5724(2) | 3188(2) | 2700(1) | 59(1) |
| O(2) | 5057(2) | 3854(2) | 3219(2) | 60(1) |
| O(3) | 9029(2) | 6525(2) | -691(2) | 61(1) |
| O(4) | 8291(2) | 6824(2) | -1364(2) | 66(1) |
| O(5) | 932(3) | 6556(3) | -782(2) | 79(2) |
| O(6) | 611(5) | 5639(4) | -610(4) | 71(3) |
| O(7) | 7138(3) | 2500 | 2690(2) | 55(2) |
| O(8) | 5132(2) | 2500 | 3585(2) | 36(1) |
| C(1) | 5372(3) | 3684(3) | 2782(2) | 53(2) |
| C(2) | 5272(3) | 4128(3) | 2319(2) | 51(2) |
| C(3) | 5727(3) | 4042(3) | 1886(2) | 61(2) |
| C(4) | 5653(3) | 4433(3) | 1458(2) | 64(2) |
| C(5) | 5118(3) | 4895(3) | 1445(2) | 60(2) |
| C(6) | 4651(4) | 4992(4) | 1906(3) | 75(2) |
| C(7) | 4742(4) | 4611(4) | 2335(3) | 70(2) |
| C(8) | 5011(3) | 5299(4) | 968(3) | 62(2) |
| C(9) | 5673(3) | 5509(4) | 691(3) | 72(2) |
| C(10) | 5606(3) | 5861(4) | 232(2) | 66(2) |
| C(11) | 4854(3) | 6008(3) | 55(3) | 65(2) |
| C(12) | 4194(3) | 5798(4) | 315(3) | 73(2) |
| C(13) | 4284(3) | 5468(3) | 788(2) | 63(2) |
| C(14) | 6327(4) | 6070(4) | -61(3) | 73(2) |
| C(15) | 7009(4) | 6140(4) | 181(3) | 95(3) |
| C(16) | 7681(4) | 6316(4) | -80(3) | 82(2) |
| C(17) | 7666(3) | 6415(3) | -610(2) | 57(2) |
| C(18) | 6955(4) | 6320(3) | -883(3) | 68(2) |
| C(19) | 6293(4) | 6136(4) | -612(3) | 77(2) |
| C(20) | 8379(3) | 6617(3) | -916(3) | 58(2) |
| C(21) | 3384(4) | 5923(4) | 111(3) | 84(3) |
| C(22) | 3204(5) | 6417(5) | -170(4) | 115(3) |
| C(23) | 2398(4) | 6485(4) | -413(3) | 73(2) |
| C(24) | 1892(5) | 6021(5) | -345(3) | 99(3) |
| C(25) | 2099(5) | 5499(5) | -100(3) | 110(3) |
| C(26) | 2826(4) | 5439(6) | 123(3) | 108(3) |
| C(27) | 1062(5) | 6062(6) | -612(4) | 113(3) |
| O(9) | 5314(8) | 2500 | 333(4) | 63(3) |
| C(28) | 4808(12) | 2500 | 711(6) | 55(5) |
| N(1) | 5138(8) | 2500 | 1245(5) | 50(3) |
| C(29) | 5918(12) | 2500 | 1356(8) | 95(8) |
| C(30) | 4516(13) | 2500 | 1617(7) | 76(6) |
| O(10) | 3945(5) | 2500 | 2899(4) | 127(3) |
| O(11) | 2738(6) | 4460(6) | 2814(5) | 113(4) |
| O(12) | 10085(14) | 7500 | 644(8) | 141(8) |

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Table 3. Bond lengths [A] and angles [deg] for zn90.

| | |
|---|---|
| Zn(1)-O(5)#1 | 1.913(4) |
| Zn(1)-O(3)#2 | 1.952(4) |
| Zn(1)-O(2) | 1.972(4) |
| Zn(1)-O(8) | 1.9759(19) |
| Zn(2)-O(1) | 2.051(4) |
| Zn(2)-O(1)#3 | 2.051(4) |
| Zn(2)-O(7) | 2.061(5) |
| Zn(2)-O(8) | 2.064(4) |
| Zn(2)-O(4)#2 | 2.093(4) |
| Zn(2)-O(4)#4 | 2.093(4) |
| O(1)-C(1) | 1.247(7) |
| O(2)-C(1) | 1.285(7) |
| O(3)-C(20) | 1.269(7) |
| O(3)-Zn(1)#5 | 1.952(4) |
| O(4)-C(20) | 1.228(7) |
| O(4)-Zn(2)#5 | 2.093(4) |
| O(5)-C(27) | 1.171(11) |
| O(5)-Zn(1)#6 | 1.913(4) |
| O(6)-C(27) | 1.198(12) |
| O(8)-Zn(1)#3 | 1.9758(19) |
| C(1)-C(2) | 1.522(8) |
| C(2)-C(3) | 1.359(8) |
| C(2)-C(7) | 1.384(9) |
| C(3)-C(4) | 1.379(8) |
| C(4)-C(5) | 1.356(9) |
| C(5)-C(6) | 1.431(9) |
| C(5)-C(8) | 1.502(8) |
| C(6)-C(7) | 1.372(8) |
| C(8)-C(13) | 1.377(7) |
| C(8)-C(9) | 1.408(8) |
| C(9)-C(10) | 1.393(8) |
| C(10)-C(11) | 1.404(8) |
| C(10)-C(14) | 1.511(8) |
| C(11)-C(12) | 1.385(8) |
| C(12)-C(13) | 1.403(8) |
| C(12)-C(21) | 1.507(9) |
| C(14)-C(15) | 1.331(9) |
| C(14)-C(19) | 1.403(9) |
| C(15)-C(16) | 1.383(8) |
| C(16)-C(17) | 1.358(9) |
| C(17)-C(18) | 1.418(8) |
| C(17)-C(20) | 1.512(8) |
| C(18)-C(19) | 1.385(8) |
| C(21)-C(22) | 1.318(11) |
| C(21)-C(26) | 1.418(12) |
| C(22)-C(23) | 1.520(11) |
| C(23)-C(24) | 1.337(11) |
| C(24)-C(25) | 1.334(12) |
| C(24)-C(27) | 1.579(8) |
| C(25)-C(26) | 1.375(10) |
| O(9)-C(28) | 1.29(2) |
| C(28)-N(1) | 1.47(2) |
| N(1)-C(29) | 1.368(17) |
| N(1)-C(30) | 1.42(2) |

| | |
|---|---|
| O(5)#1-Zn(1)-O(3)#2 | 115.8(2) |
| O(5)#1-Zn(1)-O(2) | 109.38(19) |
| O(3)#2-Zn(1)-O(2) | 113.22(18) |
| O(5)#1-Zn(1)-O(8) | 109.3(2) |
| O(3)#2-Zn(1)-O(8) | 106.21(18) |
| O(2)-Zn(1)-O(8) | 102.01(18) |
| O(1)-Zn(2)-O(1)#3 | 92.8(3) |

| | |
|---|---|
| O(1)-Zn(2)-O(7) | 87.57(15) |
| O(1)#3-Zn(2)-O(7) | 87.57(15) |
| O(1)-Zn(2)-O(8) | 88.53(14) |
| O(1)#3-Zn(2)-O(8) | 88.53(14) |
| O(7)-Zn(2)-O(8) | 174.3(2) |
| O(1)-Zn(2)-O(4)#2 | 89.21(19) |
| O(1)#3-Zn(2)-O(4)#2 | 175.49(17) |
| O(7)-Zn(2)-O(4)#2 | 88.51(16) |
| O(8)-Zn(2)-O(4)#2 | 95.54(14) |
| O(1)-Zn(2)-O(4)#4 | 175.49(17) |
| O(1)#3-Zn(2)-O(4)#4 | 89.21(19) |
| O(7)-Zn(2)-O(4)#4 | 88.51(16) |
| O(8)-Zn(2)-O(4)#4 | 95.54(14) |
| O(4)#2-Zn(2)-O(4)#4 | 88.5(3) |
| C(1)-O(1)-Zn(2) | 134.9(4) |
| C(1)-O(2)-Zn(1) | 124.3(4) |
| C(20)-O(3)-Zn(1)#5 | 115.8(4) |
| C(20)-O(4)-Zn(2)#5 | 135.9(4) |
| C(27)-O(5)-Zn(1)#6 | 115.8(6) |
| Zn(1)#3-O(8)-Zn(1) | 136.4(2) |
| Zn(1)#3-O(8)-Zn(2) | 104.26(12) |
| Zn(1)-O(8)-Zn(2) | 104.26(12) |
| O(1)-C(1)-O(2) | 126.3(5) |
| O(1)-C(1)-C(2) | 117.9(5) |
| O(2)-C(1)-C(2) | 115.8(6) |
| C(3)-C(2)-C(7) | 120.2(5) |
| C(3)-C(2)-C(1) | 118.1(5) |
| C(7)-C(2)-C(1) | 121.7(5) |
| C(2)-C(3)-C(4) | 119.9(6) |
| C(5)-C(4)-C(3) | 122.1(6) |
| C(4)-C(5)-C(6) | 117.9(5) |
| C(4)-C(5)-C(8) | 121.9(6) |
| C(6)-C(5)-C(8) | 120.2(6) |
| C(7)-C(6)-C(5) | 119.5(6) |
| C(6)-C(7)-C(2) | 120.3(6) |
| C(13)-C(8)-C(9) | 118.8(5) |
| C(13)-C(8)-C(5) | 122.0(5) |
| C(9)-C(8)-C(5) | 119.3(5) |
| C(10)-C(9)-C(8) | 121.6(6) |
| C(9)-C(10)-C(11) | 117.7(5) |
| C(9)-C(10)-C(14) | 120.4(5) |
| C(11)-C(10)-C(14) | 121.9(5) |
| C(12)-C(11)-C(10) | 121.8(5) |
| C(11)-C(12)-C(13) | 118.8(5) |
| C(11)-C(12)-C(21) | 122.2(6) |
| C(13)-C(12)-C(21) | 119.0(6) |
| C(8)-C(13)-C(12) | 121.0(5) |
| C(15)-C(14)-C(19) | 118.8(6) |
| C(15)-C(14)-C(10) | 121.9(6) |
| C(19)-C(14)-C(10) | 119.0(6) |
| C(14)-C(15)-C(16) | 123.0(6) |
| C(17)-C(16)-C(15) | 120.0(6) |
| C(16)-C(17)-C(18) | 118.4(5) |
| C(16)-C(17)-C(20) | 122.4(5) |
| C(18)-C(17)-C(20) | 119.2(5) |
| C(19)-C(18)-C(17) | 120.3(6) |
| C(18)-C(19)-C(14) | 119.2(6) |
| O(4)-C(20)-O(3) | 125.4(5) |
| O(4)-C(20)-C(17) | 118.6(5) |
| O(3)-C(20)-C(17) | 115.9(5) |

| | |
|---|---|
| C(22)-C(21)-C(26) | 116.7(8) |
| C(22)-C(21)-C(12) | 123.1(8) |
| C(26)-C(21)-C(12) | 118.9(7) |
| C(21)-C(22)-C(23) | 120.7(9) |
| C(24)-C(23)-C(22) | 117.7(7) |
| C(25)-C(24)-C(23) | 121.3(8) |

| | |
|---|---|
| C(25)-C(24)-C(27) | 119.1(9) |
| C(23)-C(24)-C(27) | 119.2(9) |
| C(24)-C(25)-C(26) | 120.8(11) |
| C(25)-C(26)-C(21) | 122.2(10) |
| O(5)-C(27)-O(6) | 124.9(9) |
| O(5)-C(27)-C(24) | 112.4(10) |
| O(6)-C(27)-C(24) | 122.6(10) |
| O(9)-C(28)-N(1) | 115.0(17) |
| C(29)-N(1)-C(30) | 126.7(15) |
| C(29)-N(1)-C(28) | 124.7(16) |
| C(30)-N(1)-C(28) | 108.6(15) |

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Symmetry transformations used to generate equivalent atoms:
#1 -x+1/2,-y+1,z+1/2 #2 -x+3/2,-y+1,z+1/2
#3 x,-y+1/2,z #4 -x+3/2,y-1/2,z+1/2
#5 -x+3/2,-y+1,z-1/2 #6 -x+1/2,-y+1,z-1/2