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Suzuki Porphyrins: New Synthons for the Fabrication of Porphyrin-Containing Supramolecular Assemblies

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Experimental Section:

[5-(4',4',5',5'-Tetramethyl[1',3',2']dioxaborolan-2'-yl)- 10,20-diphenylporphinato]zinc(II) (I). A 50 ml Schlenk flask was charged with (5-bromo-10,20-diphenylporphinato)zinc(II) (60 mg, 0.10 mmol), pinacolborane (120 μl, 0.84 mmol), triethylamine (180 μl, 1.3 mmol), *trans*-dichlorobis(triphenylphosphine)palladium(II) (3 mg, 0.003 mmol) and 10 ml of 1,2-dichloroethane under nitrogen. The mixture was stirred at 90 °C for 45 min, at which point TLC showed that the (5-bromo-10,20-diphenylporphinato)zinc(II) starting material was completely consumed. The reaction was quenched with aq KCl (10 ml), washed with water, and dried over $MgSO_4$. The solvent was evaporated, and the residue taken up in CH_2Cl_2; compound I was purified by silica gel chromatography using CH_2Cl_2 as the eluant. The first band isolated corresponded to (5,15-diphenylporphinato)zinc(II), while the second band contained the porphyrylboronate complex; isolated yield = 51 mg (79 %, based on 60 mg of (5-bromo-10,20-diphenylporphinato)zinc(II)). ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.26 (s, 1 H), 9.94 (d, J = 4.57 Hz, 2 H), 9.38 (d, J = 4.46 Hz, 2 H), 9.11 (d, J = 4.60 Hz, 2 H), 9.06 (d, J

= 4.45 Hz, 2 H), 8.22 (m, 4 H), 7.77 (m, 6 H), 1.85 (s, 12 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.9, 150.4, 150.0, 149.0, 142.8, 134.6, 132.8, 132.2, 131.8, 127.5, 126.5, 120.4, 107.3, 85.3, 25.4. Vis (CH_2Cl_2), [λ_{max} (nm), (log ε ($M^{-1}cm^{-1}$)]: 411 (5.67), 540 (4.28), 571 (3.48). MS (ESI) (M + H) m/z: 652 (Calcd 652). Anal. Calcd for $C_{38}H_{31}BN_4O_2Zn$: C, 70.00; H, 4.80; N, 8.60. Found: C, 69.74; H, 4.69; N, 8.32.

[5,15-Bis(4′,4′,5′,5′-tetramethyl[1′,3′,2′]dioxaborolan-2′-yl)-10,20-diphenylporphinato]zinc(II) (II). Under an inert atmosphere, (5,15-dibromo-10,20-diphenylporphinato)zinc(II) (95 mg, 0.13 mmol), pinacolborane (390 μl, 2.6 mmol), triethylamine (460 μl, 3.3 mmol), $Pd(PPh_3)_2Cl_2$ (3 mg, 0.003 mmol) were brought together in a 50 ml Schlenk flask along with 10 ml of 1,2-dichloroethane solvent. The mixture was stirred at 90 °C for 12 h. The reaction was quenched with aq KCl (10 ml), washed with water, dried over $MgSO_4$, and evaporated. The residue was taken up in CH_2Cl_2, and purified by silica gel chromatography using CH_2Cl_2 as the eluant. Two reduction products eluted first: dehalogenated (5,15-diphenylporphinato)zinc(II) (< 5 %), followed by [5-(4′,4′,5′,5′-tetramethyl-[1′,3′,2′]dioxaborolan-2′-yl)-10,20-diphenylporphinato]zinc(II) (< 5 %). The third band contained the product diboronated (porphinato)zinc(II) complex; isolated yield = 88 mg (85 %, based on 95 mg of (5,15-dibromo-10,20-diphenylporphinato)zinc(II)). ^1H NMR (500 MHz, pyridine-d_5): δ 10.25 (d, J = 4.48 Hz, 4 H), 9.21 (d, J = 4.49 Hz, 4 H),

8.38 (m, 4 H), 7.81 (m, 6 H), 1.80 (s, 24 H). ^{13}C NMR (125 MHz, pyridine-d_5); δ 154.0, 150.6, 144.1, 135.1, 133.8, 132.6, 128.8, 127.8, 126.9, 121.0, 85.5, 25.4. Vis (CH_2Cl_2): 395 (4.58) (sh), 414 (5.64), 545 (4.20), 580 (3.78). HRMS (ESI) m/z: 777.2748 (Calcd for $C_{44}H_{42}B_2N_4O_4Zn$ (M+) 777.2761). Anal. Calcd for $C_{44}H_{42}B_2N_4O_4Zn$: C, 67.93; H, 5.45; N, 7.20. Found: C, 68.02; H, 5.53; N, 6.93.

General Conditions for the Preparation of 5-Substituted-10,20-diphenylporphyrins via Pd-Catalyzed Cross-Coupling Reactions that Employ [5-(4',4',5',5'-Tetramethyl[1',3',2']dioxaborolan-2'-yl)- 10,20-diphenylporphinato]zinc(II) as the Transmetalating Reagent. A Schlenk-style storage tube was charged with the aryl halide (0.08 mmol), $Ba(OH)_2 \cdot 8\ H_2O$ (0.08 g, 0.24 mmol), $Pd(PPh_3)_4$ (3 mg, 0.003 mmol) and 1 to 4 equivalents of [5,15-diphenyl-10-(4',4',5',5'-tetramethyl-[1',3',2']dioxaborolan-2'-yl)-porphinato]zinc(II) (I); 15 ml of freshly distilled dimethoxyethane (DME) and 0.3 ml H_2O (deoxygenated via three freeze-pump-thaw degas cycles) were added, and the solution was heated at 80 °C under an inert atmosphere. The course of the reaction was monitored by TLC; reaction times varied between 6 and 12 h depending on the nature of the organohalide substrate. At the endpoint, the reaction was quenched with H_2O and extracted with CH_2Cl_2; the CH_2Cl_2 solution was washed several times with H_2O, dried over $CaCl_2$, and evaporated. Selected characterization data with details regarding product isolation for three representative reactions are presented below.

(5-[N-(*tert*-butoxycarbonyl)-L-phenylalanin-4′-yl]-10,20-diphenylporphinato)zinc(II) (III). The porphyrinic transmetalating reagent I (102 mg, 0.16 mmol) and N-(*tert*-butoxycarbonyl)-4-iodo-L-phenylalanine (33 mg, 0.08 mmol) were reacted according to the conditions described above for 12 h, at which time the reaction was quenched and loaded onto a silica gel column packed in 9:1 CH_2Cl_2:MeOH. The first band to elute contained a trace quantity of (5,15-diphenylporphinato)zinc(II), while the second band corresponded to the product. Evaporation of solvent gave 53 mg of compound **III** (79 % yield, based on 33 mg of N-(*tert*-butoxycarbonyl)-4-iodo-L-phenylalanine. Selected characterization data: ^{1}H NMR (500 MHz, 19:1 $CDCl_3$:pyridine-d_5): δ 10.01 (s, 1 H), 9.20 (d, J = 4.04 Hz, 2 H), 8.89 (d, J = 4.05 Hz, 2 H), 8.78 (m, 4 H), 8.09 (m, 4 H), 7.99 (d, J = 6.73 Hz, 2 H), 7.61 (m, 6 H), 7.49 (d, J = 6.63 Hz, 2 H), 5.66 (s, 1 H), 4.81 (s, 1 H), 3.51 (s, 1 H), 3.35 (s, 1 H), 1.40 (s, 9 H). ^{13}C NMR (125 MHz, 19:1 $CDCl_3$:pyridine-d_5) δ 174.6, 155.3, 149.9, 149.8, 149.6, 149.5, 143.3, 134.6, 134.4, 132.0, 131.6, 13.3, 131.2, 137.3, 127.0, 126.2, 119.8, 105.1, 79.3, 54.9, 38.5, 29.9, 29.6, 28.3. Vis (CH_2Cl_2): 413 (5.64), 541 (4.27). Anal. Calcd for $C_{46}H_{37}N_5O_4Zn$: C, 70.00; H, 4.73; N, 8.88. Found: C, 69.80; H, 4.67; N, 8.66.

3,6-Bis[10′,20′-diphenylporphinato(zinc)(II)-5′-yl]-9-*H*-carbazole (IV). Compound **I** (129 mg, 0.2 mmol) and 3,6-dibromocarbazole (16 mg, 0.05 mmol) were brought together with the appropriate quantities of base, catalyst, DME, and water and reacted for 12 h. The product was purified by column chromatography using CH_2Cl_2 as the eluant. The first band to elute contained a small quantity of (5,15-diphenylpophinato)zinc(II); the second band contained 46 mg of compound **IV**

(isolated yield = 76 % based on 16 mg 3,6-dibromocarbazole). Selected characterization data: ^{1}H NMR (500 MHz, 19:1 $CDCl_3$: pyridine-d_5): δ 10.39 (s, 1 H), 10.01 (s, 2 H), 9.22 (d, J = 4.45 Hz, 4 H), 8.95 (d, J = 4.53 Hz, 4 H), 8.91 (d, J = 4.41 Hz, 4 H), 8.87 (d, J=1.25 Hz, 2 H), 8.82 (d, J = 4.53 Hz, 4 H), 8.30 (dd, J_1 = 3.94 Hz, J_2 = 1.52 Hz, 2 H), 8.16 (d, J = 6.17 Hz, 4 H), 8.09 (d, J = 7.04 Hz, 4 H), 7.82 (d, J = 8.36 Hz, 2 H), 7.66 (m, 6 H), 7.62 (m, 6 H). ^{13}C NMR (125 MHz, 19:1 $CDCl_3$:pyridine-d_5): δ 150.3, 149.82, 149.76, 149.6, 143.39, 140.1, 134.8, 135.6, 132.9, 131.96, 131.2, 131.1, 126.9, 126.5, 126.2, 123.3, 121.9, 121.8, 119.5, 108.2, 104.9.). Vis (CH_2Cl_2): 412 (5.87), 419 (sh) (5.73), 506 (sh) (3.80), 541 (4.64), 580 (3.77). HRMS (ESI) m/z: 1213.2516 (Calcd for $C_{76}H_{45}N_9Zn$ (M+): 1213.2537. Anal. Calcd for $C_{76}H_{45}NZn_2$: C, 75.00; H, 3.90; N, 10.36. Found: C, 74.76; H, 3.80; N, 10.18.

[5-(8'-(2'',5''-Dimethoxyphenyl)naphthyl)-10,20-diphenylporphinato]zinc(II) (V). The porphyrinic transmetalating reagent I (50 mg, 0.08 mmol) and 1-(2',5'-dimethoxyphenyl)-8-iodonaphthalene (31 mg, 0.08 mmol) were reacted according to the conditions described above for 6 h, at which time the mixture was quenched and the crude product loaded onto a silica gel column packed in 19:1 hexanes:THF. During the chromatographic separation, the eluant polarity was gradually increased to 9:1 hexanes:THF; a small fraction quantity of (5,15-diphenylporphinato)zinc(II) eluted prior to the main product fraction. After evaporation of the volatiles, the product was washed with hexanes to give 45 mg of pure [5-(8'-(2'',5''-dimethoxyphenyl)naphthyl)-10,20-diphenylporphyrinato]zinc(II) (74 %, based on 50

mg of the porphyrylboronate starting material). It is worthy of note that analogous reactions carried out using a slight molar excess (e.g., 1.5 eq) of the [5-(4',4',5',5'-tetramethyl[1',3',2']dioxaborolan-2'-yl)- 10,20-diphenylporphinato]zinc(II) transmetalating reagent provide yields of compound V in excess of 90 %. ^{1}H NMR (250 MHz, $CDCl_3$): δ 10.19 (s, 1H), 9.37 (m, 2H), 9.05 (m, 2H), 8.91 (d, 1H, J = 4.6), 8.78 (m, 2H), 8.56 (d, 1H, J = 6.8), 8.47 (d, 1H, J = 6.6), 8.43 (d, 1H, J = 4.6), 8.34 (m, 2H), 8.13 (d, 1H, J = 8.2), 8.07 (d, 2H, J = 6.9), 7.89 (t, 1H, J = 7.6), 7.74 (m, 6H), 7.45 (t, 1H, J = 7.6), 6.71 (d, 1H, J = 7.0), 4.88 (d, 1H, J = 3.1), 3.13 (d, 1H, J = 9.0), 2.42 (dd, 1H, J_1 = 8.9; J_2 = 3.1), 2.20 (s, 3H), 1.26 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.15, 150.09, 149.95, 149.70, 149.64, 149.55, 149.50, 149.26, 147.96, 142.97, 142.91, 139.34, 137.48, 135.59, 134.59, 134.51, 134.40, 133.97, 133.32, 133.25, 132.31, 132.07, 132.05, 131.77, 131.72, 131.43, 131.19, 130.86, 130.36, 130.28, 129.67, 128.79, 127.57, 127.40, 127.34, 126.72, 126.57, 126.48, 125.29, 123.26, 121.22, 120.82, 119.63, 114.90, 105.93, 105.53, 104.75, 53.59, 52.45. Vis (CH_2Cl_2): 423 (5.42), 549 (4.23). HRMS (FAB) m/z: 786.1996 (Calcd for $C_{50}H_{34}N_4O_2Zn$ (M+): 786.1973).

X-ray Crystallography. The crystal structure for compound I was solved by standard heavy atom Patterson methods followed by weighted Fourier syntheses, while that for compound II was deciphered using direct methods (SIR92).[1] Tables I contain details of the crystal and data collection parameters. Both structures were determined by Dr. Patrick Carroll at the Chemistry Department's X-ray facility at the

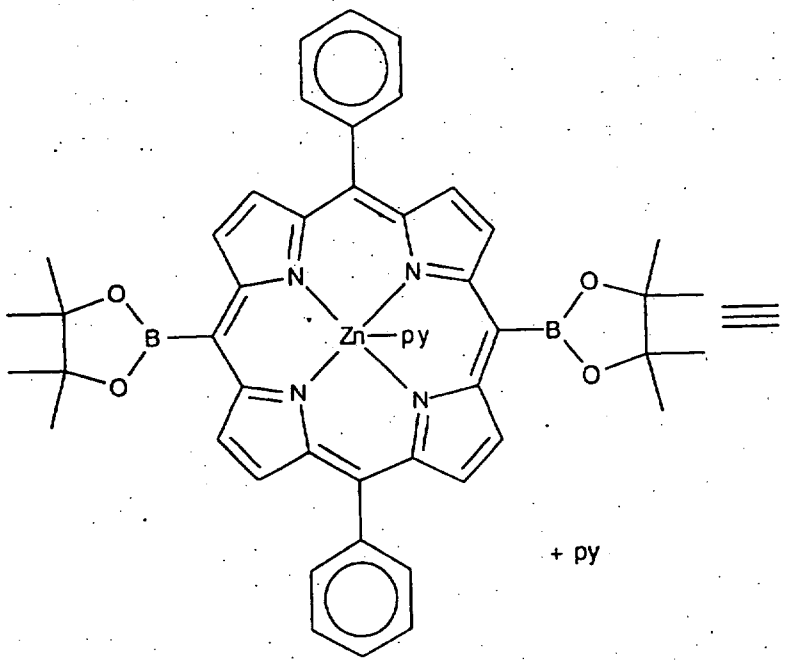
University of Pennsylvania.

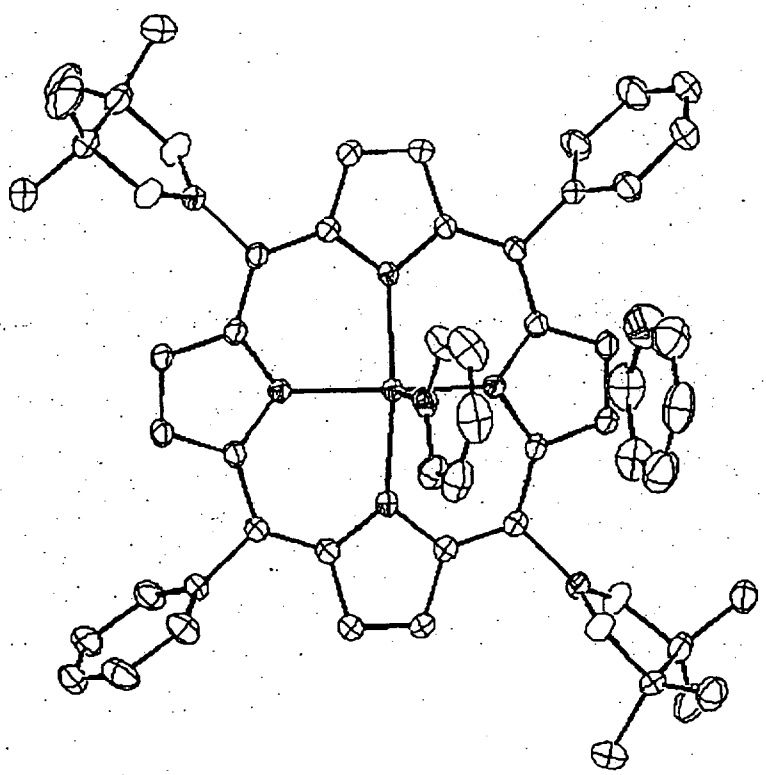
Compounds **I** and **II** crystallized forming rectangular purple plates over a period of several days. X-ray quality crystals for compound **I** were obtained by slow evaporation of a saturated benzene solution of [5-(4',4',5',5'-tetramethyl-[1',3',2']dioxaborolan-2'-yl)- 10,20-diphenylporphinato]zinc(II). The crystal dimensions were 0.42 x 0.34 x 0.03 mm. X-ray quality crystals for compound **II** were acquired by diffusing pentane vapor into a 5:1 CH_2Cl_2:pyridine solution of [5,15-bis(4',4',5',5'-tetramethyl-[1',3',2']dioxaborolan-2'-yl)-10,20-diphenylporphinato]zinc(II). The crystal dimensions were 0.42 x 0.18 x 0.06 mm.

References:

(1) Altomare, A., Burla, M. C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidoro, G. *J. Appl. Cryst.*, **1994**, *27*, 435.

X-ray Structure Determination of Compound 920





Compound 920, $ZnC_{54}B_2H_{52}N_6O_4$, crystallizes in the orthorhombic space group $P2_12_12_1$ (systematic absences h00: h=odd, 0k0: k=odd, and 00l: l=odd) with a=14.4840(10)Å, b=28.0790(13)Å, c=11.7281(7)Å, V=4769.8(5)Å^3, Z=4 and d_{calc}=1.303 g/cm^3. X-ray intensity data were collected on an Rigaku R-AXIS IIc area detector employing graphite-monochromated Mo-K_α radiation (λ=0.71069 Å) at a temperature of 180°K. Indexing was performed from a series of 1° oscillation images with exposures of 4 minutes per frame. A hemispere of data was collected using 5° oscillation angles with exposures of 12 minutes per frame and a crystal-to-detector distance of 82 mm. Oscillation images were processed using bioteX[1], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the teXsan[2] program package for further processing and structure solution on a Silicon Graphics Indigo R4000 computer. A total of 25701 reflections were measured over the ranges: $5.18 \leq 2\theta \leq 50.66°$, $-17 \leq h \leq 17$, $-33 \leq k \leq 33$, $-13 \leq l \leq 13$ yielding 8577 unique reflections (R_{int} = 0.0441). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by direct methods (SIR92[3]). It was found that there was a molecule of pyridine in the unit cell present as solvent of crystallization. Refinement was by full-matrix least squares based on F^2 using SHELXL-93[4]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by F^2 = 0). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ 0.0381P^2 + 3.7379P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a "riding" model. Refinement converged to R_1=0.0456 and wR_2=0.1010 for 8058 reflections for which $F > 4\sigma(F)$ and R_1=0.0501, wR_2=0.1048 and GOF = 1.104 for all 8577 unique, non-zero reflections and 613 variables[5]. The maximum Δ/σ in the final cycle of least squares was -0.003 and the two most prominent peaks in the final difference Fourier were +0.237 and -0.602 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Table 2. Anisotropic thermal parameters are in Table 3. Tables 4. and 5. list bond distances and bond angles. Figure 1. is an ORTEP[6] representation of the molecule with 30% probability thermal ellipsoids displayed.

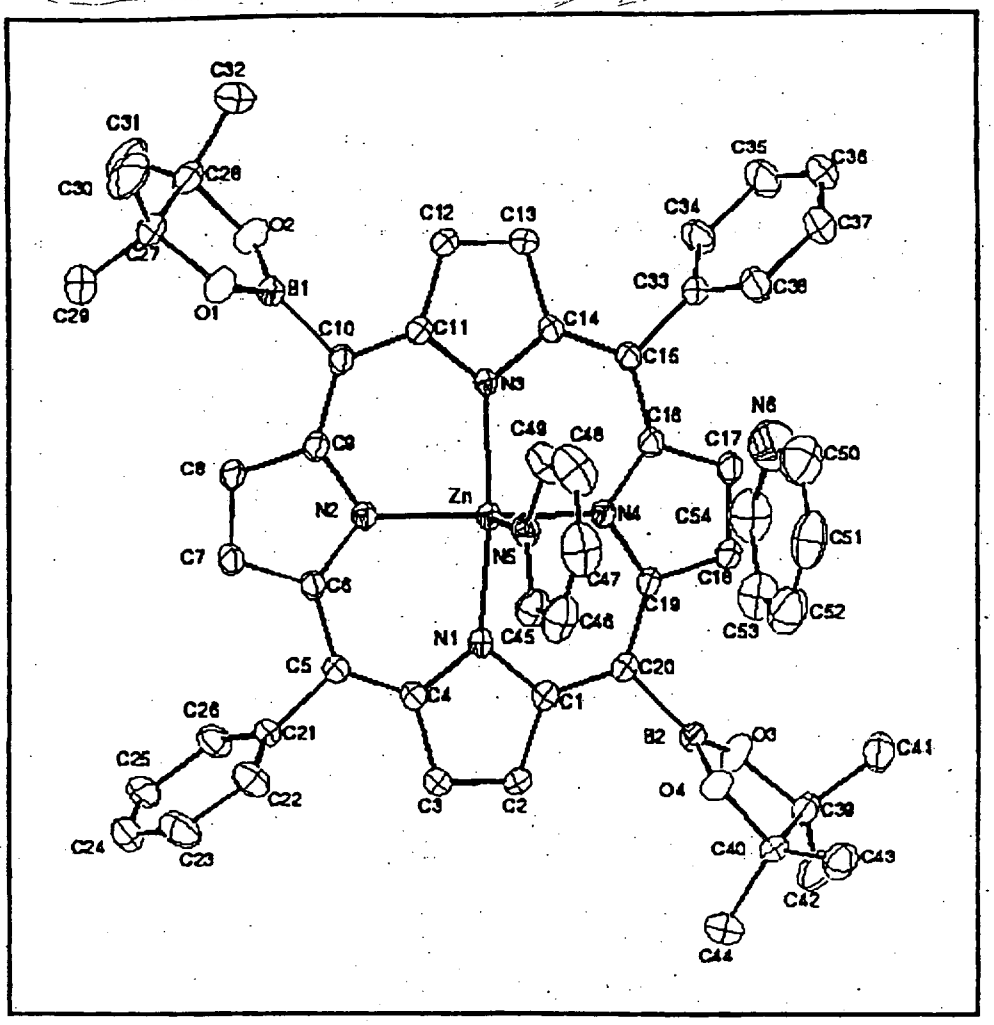


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

References

1. bioteX: A suite of Programs for the Collection, Reduction and Interpretation of Imaging Plate Data, Molecular Structure Corporation (1995).

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

3. SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidoro, G. (1994). *J. Appl. Cryst.*, **27**, 435.

4. SHELXL-93: Program for the Refinement of Crystal Structures, Sheldrick, G.M. (1993), University of Göttingen, Germany.

5. $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$

$$wR_2 = \{ \sum w (F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2 \}^{1/2}$$

GOF = $\{ \sum w (F_o^2 - F_c^2)^2 / (n - p) \}^{1/2}$ where n = the number of reflections and p = the number of parameters refined.

6. "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

Table 1. Summary of Structure Determination of Compound 920

Formula:	$ZnC_{54}B_2H_{52}N_6O_4$
Formula weight:	936.01
Crystal class:	orthorhombic
Space group:	$P2_12_12_1$ (#19)
Z	4
Cell constants:	
a	14.4840(10)Å
b	28.0790(13)Å
c	11.7281(7)Å
V	4769.8(5)Å^3
μ	5.68 cm^{-1}
crystal size, mm	0.42 x 0.18 x 0.06
D_{calc}	1.303 g/cm^3
F(000)	1960
Radiation:	Mo-K_α (λ=0.71069Å)
2θ range	5.18–50.66°
hkl collected:	-17≤ h ≤17; -33≤ k ≤33; -13≤ l ≤13
No. reflections measured:	25701
No. unique reflections:	8577 (R_{int}=0.0441)
No. observed reflections	8058 (F>4σ)
No. reflections used in refinement	8577
No. parameters	613
R indices (F>4σ)	R_1=0.0456
	wR_2=0.1010
R indices (all data)	R1=0.0501
	wR2=0.1048
GOF:	1.104
Final Difference Peaks, e/Å^3	+0.237, -0.602

Table 2. Refined Positional Parameters for Compound 920

Atom	x	y	z	U_{eq}, $Å^2$
Zn	0.46611(2)	0.614170(13)	0.66261(3)	0.03183(10)
O1	0.8336(2)	0.68681(10)	0.7991(2)	0.0487(7)
O2	0.8031(2)	0.63188(12)	0.9367(2)	0.0585(8)
O3	0.1150(2)	0.51217(9)	0.5368(3)	0.0491(7)
O4	0.1378(2)	0.57580(10)	0.4191(2)	0.0487(7)
N1	0.4335(2)	0.57171(10)	0.5231(3)	0.0355(6)
N2	0.6029(2)	0.60761(10)	0.6179(2)	0.0327(6)
N3	0.5079(2)	0.63563(10)	0.8249(2)	0.0330(6)
N4	0.3386(2)	0.59805(10)	0.7311(2)	0.0319(6)
N5	0.4337(2)	0.68283(11)	0.5887(3)	0.0414(7)
C1	0.3460(2)	0.55721(13)	0.4928(3)	0.0383(8)
C2	0.3492(3)	0.53600(14)	0.3806(3)	0.0449(9)
H2	0.2991(3)	0.52405(14)	0.3402(3)	0.060
C3	0.4373(2)	0.53657(14)	0.3450(4)	0.0458(9)
H3	0.4596(2)	0.52465(14)	0.2764(4)	0.061
C4	0.4908(2)	0.55932(13)	0.4340(3)	0.0369(8)
C5	0.5854(2)	0.56731(13)	0.4303(3)	0.0348(8)
C6	0.6371(2)	0.59021(12)	0.5158(3)	0.0340(7)
C7	0.7340(2)	0.59882(13)	0.5097(3)	0.0393(8)
H7	0.7728(2)	0.59098(13)	0.4494(3)	0.052
C8	0.7590(2)	0.62053(13)	0.6079(3)	0.0382(8)
H8	0.8183(2)	0.63016(13)	0.6277(3)	0.051
C9	0.6771(2)	0.62601(11)	0.6762(3)	0.0346(7)
C10	0.6758(2)	0.64388(13)	0.7885(3)	0.0351(7)

C11	0.5963(2)	0.64727(12)	0.8570(3)	0.0334(7)
C12	0.5949(2)	0.66435(14)	0.9736(3)	0.0394(8)
H12	0.6457(2)	0.67459(14)	1.0156(3)	0.052
C13	0.5069(2)	0.66272(13)	1.0102(3)	0.0402(8)
H13	0.4856(2)	0.67170(13)	1.0818(3)	0.053
C14	0.4520(2)	0.64438(12)	0.9173(3)	0.0327(7)
C15	0.3576(2)	0.63402(13)	0.9233(3)	0.0345(7)
C16	0.3066(2)	0.61049(12)	0.8379(3)	0.0338(6)
C17	0.2127(2)	0.59537(13)	0.8499(3)	0.0419(8)
H17	0.1760(2)	0.59858(13)	0.9145(3)	0.056
C18	0.1875(2)	0.57545(13)	0.7492(3)	0.0384(8)
H18	0.1298(2)	0.56289(13)	0.7316(3)	0.051
C19	0.2665(2)	0.57735(12)	0.6745(3)	0.0345(7)
C20	0.2673(2)	0.56058(12)	0.5612(3)	0.0356(8)
C21	0.6396(2)	0.54921(12)	0.3303(3)	0.0364(7)
C22	0.6494(3)	0.5757(2)	0.2313(3)	0.0530(10)
H22	0.6158(3)	0.6036(2)	0.2219(3)	0.070
C23	0.7098(4)	0.5606(2)	0.1457(4)	0.0687(13)
H23	0.7168(4)	0.5786(2)	0.0796(4)	0.091
C24	0.7587(3)	0.5192(2)	0.1593(4)	0.0643(12)
H24	0.7999(3)	0.5096(2)	0.1030(4)	0.086
C25	0.7475(3)	0.4923(2)	0.2539(4)	0.0620(12)
H25	0.7802(3)	0.4639(2)	0.2616(4)	0.083
C26	0.6872(3)	0.50669(14)	0.3401(4)	0.0513(9)
H26	0.6791(3)	0.48769(14)	0.4043(4)	0.068
C27	0.9216(3)	0.6788(2)	0.8592(4)	0.0506(10)
C28	0.8905(3)	0.6538(2)	0.9708(4)	0.0591(12)

C29	0.9783(3)	0.6478(2)	0.7828(4)	0.0705(13)
H29a	0.951(2)	0.6167(4)	0.779(3)	0.106
H29b	1.0398(8)	0.6453(11)	0.813(2)	0.106
H29c	0.980(2)	0.6615(7)	0.7078(9)	0.106
C30	0.9681(4)	0.7262(2)	0.8769(5)	0.091(2)
H30a	1.024(2)	0.7217(3)	0.919(4)	0.137
H30b	0.9278(14)	0.7469(6)	0.919(4)	0.137
H30c	0.982(3)	0.7403(8)	0.8042(5)	0.137
C31	0.9546(3)	0.6135(2)	1.0111(5)	0.093(2)
H31a	1.0123(13)	0.6268(3)	1.036(4)	0.140
H31b	0.966(3)	0.5918(10)	0.9492(13)	0.140
H31c	0.926(2)	0.5967(11)	1.073(3)	0.140
C32	0.8676(4)	0.6874(3)	1.0680(5)	0.112(3)
H32a	0.9218(11)	0.705(2)	1.089(3)	0.167
H32b	0.846(4)	0.6694(3)	1.132(2)	0.167
H32c	0.820(3)	0.7091(13)	1.044(2)	0.167
C33	0.3051(2)	0.64866(13)	1.0277(3)	0.0377(8)
C34	0.3113(3)	0.6239(2)	1.1293(3)	0.0534(11)
H34	0.3507(3)	0.5978(2)	1.1344(3)	0.071
C35	0.2593(3)	0.6374(2)	1.2243(4)	0.0662(13)
H35	0.2632(3)	0.6201(2)	1.2918(4)	0.088
C36	0.2028(3)	0.6761(2)	1.2172(4)	0.0633(13)
H36	0.1685(3)	0.6853(2)	1.2805(4)	0.084
C37	0.1960(3)	0.7018(2)	1.1165(4)	0.0618(12)
H37	0.1572(3)	0.7281(2)	1.1122(4)	0.082
C38	0.2476(3)	0.6878(2)	1.0218(4)	0.0505(10)
H38	0.2434(3)	0.7050(2)	0.9542(4)	0.067

C39	0.0256(3)	0.52012(13)	0.4776(3)	0.0449(8)
C40	0.0544(2)	0.5525(2)	0.3758(3)	0.0453(9)
C41	-0.0354(3)	0.5446(2)	0.5637(4)	0.0571(10)
H41a	-0.0963(7)	0.5482(10)	0.5326(11)	0.086
H41b	-0.0104(12)	0.5753(5)	0.581(2)	0.086
H41c	-0.038(2)	0.5257(6)	0.6319(11)	0.086
C42	-0.0123(3)	0.4726(2)	0.4431(5)	0.073(2)
H42a	-0.068(2)	0.4771(2)	0.400(3)	0.110
H42b	-0.026(3)	0.4543(6)	0.5100(5)	0.110
H42c	0.0326(11)	0.4561(6)	0.398(3)	0.110
C43	-0.0148(3)	0.5912(2)	0.3462(4)	0.0633(11)
H43a	-0.0714(8)	0.5769(2)	0.321(3)	0.095
H43b	0.0097(10)	0.6109(8)	0.287(2)	0.095
H43c	-0.027(2)	0.6104(8)	0.4125(9)	0.095
C44	0.0824(4)	0.5255(2)	0.2686(4)	0.079(2)
H44a	0.0292(6)	0.5103(12)	0.236(2)	0.119
H44b	0.128(2)	0.5017(10)	0.2879(7)	0.119
H44c	0.108(3)	0.5473(3)	0.2143(14)	0.119
C45	0.4061(3)	0.6865(2)	0.4792(4)	0.0537(10)
H45	0.4041(3)	0.6592(2)	0.4342(4)	0.071
C46	0.3808(3)	0.7300(2)	0.4321(5)	0.071(2)
H46	0.3624(3)	0.7319(2)	0.3562(5)	0.094
C47	0.3832(4)	0.7697(2)	0.4983(6)	0.083(2)
H47	0.3664(4)	0.7991(2)	0.4682(6)	0.111
C48	0.4108(4)	0.7662(2)	0.6098(5)	0.082(2)
H48	0.4129(4)	0.7930(2)	0.6561(5)	0.109
C49	0.4353(3)	0.72192(14)	0.6525(4)	0.0606(11)

H49	0.4536(3)	0.71952(14)	0.7283(4)	0.081
N6	0.1988(4)	0.7459(2)	0.7465(5)	0.101(2)
C50	0.1700(4)	0.7860(2)	0.6980(6)	0.091(2)
H50	0.1702(4)	0.8139(2)	0.7409(6)	0.121
C51	0.1403(4)	0.7880(2)	0.5881(7)	0.089(2)
H51	0.1212(4)	0.8170(2)	0.5579(7)	0.118
C52	0.1382(4)	0.7479(3)	0.5207(6)	0.089(2)
H52	0.1184(4)	0.7491(3)	0.4453(6)	0.119
C53	0.1667(4)	0.7059(2)	0.5706(6)	0.086(2)
H53	0.1655(4)	0.6775(2)	0.5297(6)	0.115
C54	0.1966(4)	0.7067(2)	0.6806(6)	0.083(2)
H54	0.2169(4)	0.6782(2)	0.7124(6)	0.111
B1	0.7716(3)	0.65527(14)	0.8432(4)	0.0385(8)
B2	0.1722(3)	0.54808(14)	0.5051(4)	0.0357(9)

$U_{eq} = {}^{1}/_{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Refined Thermal Parameters (U's) for Compound 920

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Zn	0.0259(2)	0.0397(2)	0.0299(2)	-0.0001(2)	0.0015(2)	-0.0012(2)
O1	0.0339(14)	0.056(2)	0.056(2)	0.0023(13)	-0.0043(12)	-0.0095(12)
O2	0.039(2)	0.090(2)	0.047(2)	0.017(2)	-0.0089(12)	-0.0138(14)
O3	0.0324(13)	0.049(2)	0.066(2)	0.0036(13)	-0.0128(13)	-0.0047(12)
O4	0.0366(14)	0.064(2)	0.045(2)	0.0054(13)	-0.0064(12)	-0.0094(13)
N1	0.0270(14)	0.042(2)	0.037(2)	-0.0015(13)	0.0037(12)	-0.0001(12)
N2	0.0280(13)	0.040(2)	0.0304(13)	0.0011(12)	0.0009(11)	-0.0005(12)
N3	0.0258(13)	0.0431(14)	0.030(2)	-0.0001(13)	0.0039(11)	-0.0025(10)
N4	0.0265(14)	0.038(2)	0.031(2)	0.0014(11)	-0.0003(11)	-0.0020(11)
N5	0.038(2)	0.046(2)	0.040(2)	0.0069(14)	0.0067(13)	0.0021(13)
C1	0.031(2)	0.044(2)	0.039(2)	-0.006(2)	-0.002(2)	0.001(2)
C2	0.033(2)	0.058(2)	0.043(2)	-0.015(2)	-0.006(2)	-0.001(2)
C3	0.037(2)	0.060(2)	0.041(2)	-0.014(2)	0.002(2)	0.001(2)
C4	0.035(2)	0.043(2)	0.033(2)	-0.003(2)	0.0013(14)	0.0044(14)
C5	0.033(2)	0.040(2)	0.031(2)	-0.002(2)	-0.0001(14)	0.0034(14)
C6	0.029(2)	0.039(2)	0.034(2)	0.001(2)	0.0043(14)	0.0022(14)
C7	0.029(2)	0.052(2)	0.037(2)	0.003(2)	0.007(2)	0.001(2)
C8	0.025(2)	0.048(2)	0.041(2)	0.002(2)	0.0052(14)	-0.006(2)
C9	0.027(2)	0.042(2)	0.035(2)	0.002(2)	-0.0001(14)	-0.0020(13)
C10	0.026(2)	0.042(2)	0.037(2)	0.002(2)	0.0020(14)	-0.0021(14)
C11	0.028(2)	0.041(2)	0.031(2)	-0.0015(14)	-0.0009(14)	-0.0025(13)
C12	0.031(2)	0.052(2)	0.035(2)	-0.003(2)	-0.003(2)	-0.003(2)
C13	0.037(2)	0.052(2)	0.031(2)	-0.006(2)	-0.0008(14)	0.001(2)
C14	0.029(2)	0.041(2)	0.028(2)	-0.0003(14)	0.0026(13)	0.0012(14)

C15	0.031(2)	0.045(2)	0.028(2)	0.0018(14)	0.0033(14)	0.0023(14)
C16	0.030(2)	0.041(2)	0.030(2)	0.002(2)	0.0020(14)	0.0014(14)
C17	0.028(2)	0.058(2)	0.039(2)	0.006(2)	0.006(2)	0.000(2)
C18	0.026(2)	0.048(2)	0.041(2)	-0.001(2)	0.001(2)	-0.005(2)
C19	0.024(2)	0.042(2)	0.038(2)	0.000(2)	-0.001(2)	-0.0013(13)
C20	0.028(2)	0.041(2)	0.038(2)	-0.001(2)	-0.0038(14)	-0.0017(14)
C21	0.032(2)	0.044(2)	0.033(2)	-0.007(2)	0.004(2)	0.0008(14)
C22	0.067(3)	0.051(2)	0.040(2)	0.004(2)	0.013(2)	0.003(2)
C23	0.086(3)	0.075(3)	0.044(3)	-0.005(2)	0.022(2)	-0.013(3)
C24	0.049(2)	0.085(3)	0.058(3)	-0.026(3)	0.015(2)	-0.003(2)
C25	0.060(3)	0.072(3)	0.054(3)	-0.023(2)	-0.002(2)	0.022(2)
C26	0.063(2)	0.053(2)	0.039(2)	-0.003(2)	-0.002(2)	0.020(2)
C27	0.033(2)	0.063(2)	0.057(3)	-0.008(2)	-0.002(2)	-0.009(2)
C28	0.034(2)	0.095(3)	0.048(2)	0.000(2)	-0.008(2)	-0.010(2)
C29	0.043(2)	0.104(4)	0.064(3)	-0.011(3)	0.004(2)	0.006(3)
C30	0.057(3)	0.083(3)	0.133(5)	-0.018(3)	-0.017(3)	-0.030(3)
C31	0.050(3)	0.148(5)	0.082(4)	0.041(4)	-0.025(3)	-0.007(4)
C32	0.067(4)	0.194(8)	0.073(4)	-0.068(5)	0.000(3)	-0.011(4)
C33	0.029(2)	0.051(2)	0.033(2)	-0.004(2)	0.0013(14)	0.000(2)
C34	0.056(2)	0.070(3)	0.034(2)	0.006(2)	0.010(2)	0.007(2)
C35	0.068(3)	0.092(4)	0.039(2)	0.005(2)	0.015(2)	0.006(3)
C36	0.052(3)	0.093(4)	0.046(3)	-0.016(2)	0.014(2)	-0.001(2)
C37	0.055(3)	0.068(3)	0.063(3)	-0.015(2)	0.010(2)	0.011(2)
C38	0.047(2)	0.061(3)	0.044(2)	0.000(2)	0.007(2)	0.010(2)
C39	0.031(2)	0.048(2)	0.056(2)	-0.003(2)	-0.008(2)	0.000(2)
C40	0.035(2)	0.061(2)	0.040(2)	-0.008(2)	-0.006(2)	0.000(2)
C41	0.041(2)	0.072(3)	0.058(2)	0.003(2)	0.003(2)	0.006(2)

C42	0.053(3)	0.055(3)	0.111(4)	-0.013(3)	-0.021(3)	-0.012(2)
C43	0.053(3)	0.076(3)	0.061(3)	0.012(2)	-0.008(2)	0.006(2)
C44	0.072(3)	0.108(4)	0.057(3)	-0.034(3)	-0.002(3)	0.001(3)
C45	0.046(2)	0.062(3)	0.053(3)	0.017(2)	-0.005(2)	-0.001(2)
C46	0.053(3)	0.086(4)	0.074(3)	0.041(3)	-0.007(2)	0.007(3)
C47	0.084(4)	0.062(3)	0.104(5)	0.039(3)	0.016(3)	0.021(3)
C48	0.110(5)	0.044(3)	0.093(4)	0.006(3)	0.028(3)	0.013(3)
C49	0.079(3)	0.049(2)	0.054(3)	0.009(2)	0.013(2)	0.011(2)
N6	0.103(4)	0.108(4)	0.092(4)	0.017(3)	0.000(3)	0.016(3)
C50	0.084(4)	0.077(4)	0.112(5)	-0.004(3)	-0.002(4)	0.005(3)
C51	0.058(3)	0.077(4)	0.133(6)	0.038(4)	0.005(4)	0.007(3)
C52	0.067(4)	0.118(5)	0.082(4)	0.023(4)	-0.008(3)	0.003(3)
C53	0.075(4)	0.090(4)	0.093(4)	0.001(3)	0.002(3)	0.002(3)
C54	0.080(4)	0.064(3)	0.106(5)	0.032(3)	0.011(4)	0.007(3)
B1	0.030(2)	0.050(2)	0.036(2)	-0.003(2)	0.002(2)	-0.003(2)
B2	0.030(2)	0.038(2)	0.039(2)	-0.005(2)	-0.002(2)	0.000(2)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+ 2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$.

Table 4. Bond Distances in Compound 920, Å

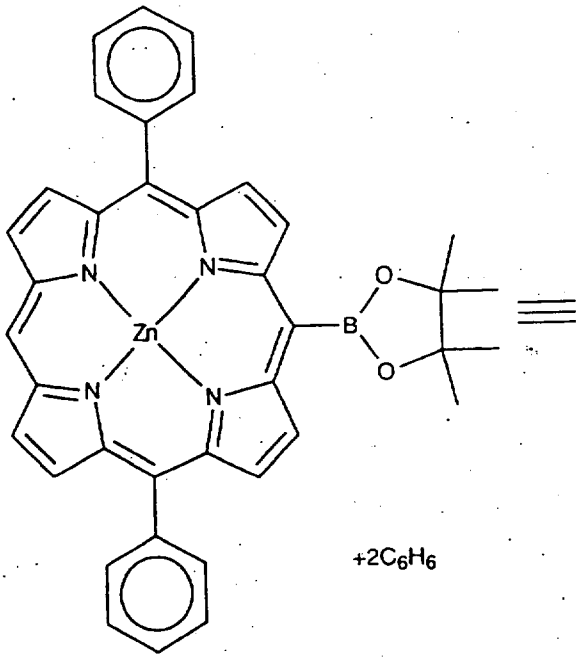
Zn-N2	2.057(3)	Zn-N4	2.064(3)	Zn-N1	2.079(3)
Zn-N3	2.086(3)	Zn-N5	2.165(3)	O1-B1	1.363(5)
O1-C27	1.473(5)	O2-B1	1.358(5)	O2-C28	1.464(5)
O3-B2	1.357(5)	O3-C39	1.486(4)	O4-B2	1.368(5)
O4-C40	1.465(4)	N1-C1	1.377(4)	N1-C4	1.379(4)
N2-C9	1.375(4)	N2-C6	1.385(4)	N3-C11	1.374(4)
N3-C14	1.375(4)	N4-C19	1.367(4)	N4-C16	1.381(4)
N5-C49	1.329(5)	N5-C45	1.349(5)	C1-C20	1.397(5)
C1-C2	1.445(5)	C2-C3	1.342(5)	C3-C4	1.448(5)
C4-C5	1.389(5)	C5-C6	1.407(5)	C5-C21	1.501(5)
C6-C7	1.426(5)	C7-C8	1.353(5)	C8-C9	1.440(4)
C9-C10	1.409(5)	C10-C11	1.407(5)	C10-B1	1.562(5)
C11-C12	1.449(5)	C12-C13	1.345(5)	C13-C14	1.445(5)
C14-C15	1.400(5)	C15-C16	1.409(5)	C15-C33	1.499(5)
C16-C17	1.432(4)	C17-C18	1.357(5)	C18-C19	1.443(5)
C19-C20	1.410(5)	C20-B2	1.567(5)	C21-C26	1.384(5)
C21-C22	1.386(5)	C22-C23	1.398(6)	C23-C24	1.370(7)
C24-C25	1.354(7)	C25-C26	1.396(6)	C27-C29	1.495(6)
C27-C30	1.507(6)	C27-C28	1.552(6)	C28-C32	1.516(7)
C28-C31	1.539(7)	C33-C38	1.381(5)	C33-C34	1.382(5)
C34-C35	1.397(6)	C35-C36	1.363(7)	C36-C37	1.387(7)
C37-C38	1.396(6)	C39-C42	1.497(5)	C39-C41	1.507(6)
C39-C40	1.558(5)	C40-C43	1.519(5)	C40-C44	1.524(6)
C45-C46	1.391(6)	C46-C47	1.361(8)	C47-C48	1.371(8)
C48-C49	1.385(6)	N6-C50	1.330(8)	N6-C54	1.344(8)
C50-C51	1.360(9)	C51-C52	1.376(9)	C52-C53	1.381(9)
C53-C54	1.361(9)				

Table 5. Bond Angles in Compound 920, °

N2-Zn-N4	160.21(11)	N2-Zn-N1	88.10(11)	N4-Zn-N1	88.69(11)
N2-Zn-N3	88.80(10)	N4-Zn-N3	88.16(10)	N1-Zn-N3	161.74(11)
N2-Zn-N5	100.76(11)	N4-Zn-N5	99.03(11)	N1-Zn-N5	98.41(12)
N3-Zn-N5	99.85(11)	B1-O1-C27	106.9(3)	B1-O2-C28	107.9(3)
B2-O3-C39	107.0(3)	B2-O4-C40	107.6(3)	C1-N1-C4	106.4(3)
C1-N1-Zn	125.6(2)	C4-N1-Zn	127.2(2)	C9-N2-C6	106.4(3)
C9-N2-Zn	126.3(2)	C6-N2-Zn	126.6(2)	C11-N3-C14	106.9(3)
C11-N3-Zn	126.1(2)	C14-N3-Zn	126.9(2)	C19-N4-C16	106.9(3)
C19-N4-Zn	126.0(2)	C16-N4-Zn	126.7(2)	C49-N5-C45	118.6(4)
C49-N5-Zn	120.4(3)	C45-N5-Zn	120.8(3)	N1-C1-C20	125.6(3)
N1-C1-C2	109.1(3)	C20-C1-C2	125.2(3)	C3-C2-C1	108.0(3)
C2-C3-C4	106.9(3)	N1-C4-C5	125.1(3)	N1-C4-C3	109.6(3)
C5-C4-C3	125.2(3)	C4-C5-C6	125.3(3)	C4-C5-C21	119.0(3)
C6-C5-C21	115.7(3)	N2-C6-C5	125.9(3)	N2-C6-C7	109.6(3)
C5-C6-C7	124.4(3)	C8-C7-C6	107.3(3)	C7-C8-C9	107.6(3)
N2-C9-C10	126.0(3)	N2-C9-C8	109.1(3)	C10-C9-C8	124.7(3)
C11-C10-C9	124.7(3)	C11-C10-B1	118.5(3)	C9-C10-B1	116.4(3)
N3-C11-C10	126.1(3)	N3-C11-C12	108.9(3)	C10-C11-C12	125.0(3)
C13-C12-C11	107.6(3)	C12-C13-C14	107.1(3)	N3-C14-C15	125.3(3)
N3-C14-C13	109.5(3)	C15-C14-C13	125.1(3)	C14-C15-C16	125.0(3)
C14-C15-C33	118.7(3)	C16-C15-C33	116.3(3)	N4-C16-C15	126.0(3)
N4-C16-C17	109.5(3)	C15-C16-C17	124.5(3)	C18-C17-C16	107.0(3)
C17-C18-C19	107.4(3)	N4-C19-C20	126.4(3)	N4-C19-C18	109.1(3)
C20-C19-C18	124.5(3)	C1-C20-C19	124.8(3)	C1-C20-B2	117.5(3)
C19-C20-B2	117.6(3)	C26-C21-C22	118.8(4)	C26-C21-C5	119.2(3)
C22-C21-C5	121.8(3)	C21-C22-C23	120.2(4)	C24-C23-C22	119.8(4)
C25-C24-C23	120.5(4)	C24-C25-C26	120.5(4)	C21-C26-C25	120.1(4)

O1-C27-C29	106.1(3)	O1-C27-C30	108.6(4)	C29-C27-C30	110.6(4)
O1-C27-C28	102.8(3)	C29-C27-C28	113.7(4)	C30-C27-C28	114.4(4)
O2-C28-C32	106.1(4)	O2-C28-C31	107.3(4)	C32-C28-C31	111.0(5)
O2-C28-C27	102.2(3)	C32-C28-C27	114.6(5)	C31-C28-C27	114.6(4)
C38-C33-C34	118.9(4)	C38-C33-C15	118.9(3)	C34-C33-C15	122.2(3)
C33-C34-C35	121.0(4)	C36-C35-C34	119.4(4)	C35-C36-C37	120.6(4)
C36-C37-C38	119.6(4)	C33-C38-C37	120.4(4)	O3-C39-C42	108.1(3)
O3-C39-C41	105.5(3)	C42-C39-C41	111.8(4)	O3-C39-C40	102.3(3)
C42-C39-C40	114.2(4)	C41-C39-C40	113.9(3)	O4-C40-C43	107.7(3)
O4-C40-C44	106.8(3)	C43-C40-C44	110.2(4)	O4-C40-C39	102.4(3)
C43-C40-C39	114.6(3)	C44-C40-C39	114.4(4)	N5-C45-C46	121.5(5)
C47-C46-C45	119.1(5)	C46-C47-C48	119.5(5)	C47-C48-C49	119.0(5)
N5-C49-C48	122.2(5)	C50-N6-C54	116.1(6)	N6-C50-C51	122.7(6)
C50-C51-C52	121.1(6)	C51-C52-C53	116.8(6)	C54-C53-C52	118.8(6)
N6-C54-C53	124.5(5)	O2-B1-O1	113.5(3)	O2-B1-C10	122.1(3)
O1-B1-C10	124.2(4)	O3-B2-O4	113.8(3)	O3-B2-C20	126.0(3)
O4-B2-C20	120.1(3)				

X-ray Structure Determination of Compound 922



Compound 922, $C_{38}H31BN_4O_2Zn \cdot 2C_6H_6$, crystallizes in the monoclinic space group $P2_1/c$ (systematic absences 0k0: k=odd and h0l: l=odd) with a=18.5272(4)Å, b=18.3603(6)Å, c=12.3747(3)Å, β=107.3220(10)°, V=4018.5(2)Å^3, Z=4 and d_{calc}=1.336 g/cm^3. X-ray intensity data were collected on an Rigaku R-AXIS IIc area detector employing graphite-monochromated Mo-K_α radiation (λ=0.71069 Å) at a temperature of 170°K. Indexing was performed from a series of 1° oscillation images with exposures of 8 minutes per frame. A hemispere of data was collected using 5° oscillation angles with exposures of 10 minutes per frame and a crystal-to-detector distance of 82 mm. Oscillation images were processed using bioteX[1], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the teXsan[2] program package for further processing and structure solution on a Silicon Graphics Indigo R4000 computer. A total of 23673 reflections were measured over the ranges: $5.00 \le 2\theta \le 50.68°$, $-22 \le h \le 22$, $-22 \le k \le 22$, $-14 \le l \le 14$ yielding 7239 unique reflections (R_{int} = 0.0468). The intensity data were corrected for Lorentz and polarization effects but not for absorption.

The structure was solved by standard heavy atom Patterson methods followed by weighted Fourier syntheses. The unit cell was found to contain two molecules of benzene solvent per asymmetric

unit. Refinement was by full-matrix least squares based on F^2 using SHELXL-93[3]. All reflections were used during refinement (F^2's that were experimentally negative were replaced by $F^2 = 0$). The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ 0.0348P^2 + 6.4350P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined according to a "riding" model. Refinement converged to $R_1=0.0630$ and $wR_2=0.1250$ for 6378 reflections for which $F > 4\sigma(F)$ and $R_1=0.0740$, $wR_2=0.1303$ and GOF = 1.190 for all 7239 unique, non-zero reflections and 531 variables[4]. The maximum Δ/σ in the final cycle of least squares was -0.002 and the two most prominent peaks in the final difference Fourier were +0.419 and -0.576 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Table 2. Anisotropic thermal parameters are in Table 3. Tables 4. and 5. list bond distances and bond angles. Figure 1. is an ORTEP[5] representation of the molecule with 30% probability thermal ellipsoids displayed.

Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

References

1. bioteX: A suite of Programs for the Collection, Reduction and Interpretation of Imaging Plate Data, Molecular Structure Corporation (1995).

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

3. SHELXL-93: Program for the Refinement of Crystal Structures, Sheldrick, G.M. (1993), University of Göttingen, Germany.

4. $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$

$wR_2 = \{ \sum w (F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2\}^{1/2}$

$GOF = \{ \sum w (F_o^2 - F_c^2)^2 / (n - p)\}^{1/2}$ where n = the number of reflections and p = the number of parameters refined.

5. "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

Table 1. Summary of Structure Determination of Compound 922

Formula:	$C_{50}H_{43}BN_4O_2Zn$
Formula weight:	808.06
Crystal class:	monoclinic
Space group:	$P2_1/c$ (#14)
Z	4
Cell constants:	
a	18.5272(4)Å
b	18.3603(6)Å
c	12.3747(3)Å
β	107.3220(10)°
V	4018.5(2)Å^3
μ	6.58 cm^{-1}
crystal size, mm	0.42 x 0.34 x 0.03
D_{calc}	1.336 g/cm^3
F(000)	1688
Radiation:	Mo-K_α (λ=0.71069Å)
2θ range	5.00–50.68°
hkl collected:	-22≤ h ≤22; -22≤ k ≤22; -14≤ l ≤14
No. reflections measured:	23673
No. unique reflections:	7239 (R_{int}=0.0468)
No. observed reflections	6378 (F>4σ)
No. reflections used in refinement	7239
No. parameters	531
R indices (F>4σ)	R_1=0.0630
	wR_2=0.1250
R indices (all data)	R1=0.0740
	wR2=0.1303
GOF:	1.190
Final Difference Peaks, e/Å^3	+0.419, -0.576

Table 2. Refined Positional Parameters for Compound 922

Atom	x	y	z	U_{eq}, Å^2
Zn	0.14405(2)	0.01532(2)	0.07136(3)	0.02866(12)
O1	0.33551(14)	0.01006(14)	-0.2191(2)	0.0407(6)
O2	0.40551(14)	0.09368(14)	-0.0955(2)	0.0386(6)
N1	0.0947(2)	-0.0723(2)	0.1219(2)	0.0293(6)
N2	0.2120(2)	-0.0541(2)	0.0172(2)	0.0292(6)
N3	0.1960(2)	0.1022(2)	0.0249(2)	0.0289(6)
N4	0.0721(2)	0.0844(2)	0.1174(2)	0.0295(6)
C1	0.0329(2)	-0.0698(2)	0.1624(3)	0.0308(7)
C2	0.0122(2)	-0.1423(2)	0.1839(3)	0.0352(8)
H2	-0.0272(2)	-0.1552(2)	0.2122(3)	0.047
C3	0.0602(2)	-0.1885(2)	0.1556(3)	0.0341(8)
H3	0.0602(2)	-0.2391(2)	0.1608(3)	0.045
C4	0.1114(2)	-0.1449(2)	0.1158(3)	0.0291(7)
C5	0.1681(2)	-0.1720(2)	0.0722(3)	0.0294(7)
C6	0.2152(2)	-0.1287(2)	0.0278(3)	0.0296(7)
C7	0.2753(2)	-0.1566(2)	-0.0133(3)	0.0336(8)
H7	0.2897(2)	-0.2050(2)	-0.0142(3)	0.045
C8	0.3065(2)	-0.0988(2)	-0.0502(3)	0.0333(8)
H8	0.3458(2)	-0.1004(2)	-0.0826(3)	0.044
C9	0.2681(2)	-0.0343(2)	-0.0304(3)	0.0293(7)
C10	0.2885(2)	0.0371(2)	-0.0489(3)	0.0299(7)
C11	0.2572(2)	0.1002(2)	-0.0165(3)	0.0303(7)
C12	0.2834(2)	0.1732(2)	-0.0247(3)	0.0358(8)
H12	0.3240(2)	0.1866(2)	-0.0501(3)	0.048

C13	0.2381(2)	0.2187(2)	0.0113(3)	0.0328(8)
H13	0.2418(2)	0.2692(2)	0.0159(3)	0.044
C14	0.1828(2)	0.1745(2)	0.0413(3)	0.0288(7)
C15	0.1245(2)	0.2020(2)	0.0806(3)	0.0290(7)
C16	0.0723(2)	0.1590(2)	0.1146(3)	0.0284(7)
C17	0.0118(2)	0.1869(2)	0.1544(3)	0.0331(8)
H17	-0.0004(2)	0.2356(2)	0.1604(3)	0.044
C18	-0.0235(2)	0.1286(2)	0.1811(3)	0.0342(8)
H18	-0.0648(2)	0.1297(2)	0.2091(3)	0.046
C19	0.0137(2)	0.0647(2)	0.1589(3)	0.0298(7)
C20	-0.0035(2)	-0.0068(2)	0.1785(3)	0.0316(8)
H20	-0.0447(2)	-0.0132(2)	0.2060(3)	0.042
C21	0.1768(2)	-0.2529(2)	0.0658(3)	0.0306(7)
C22	0.2005(2)	-0.2964(2)	0.1620(3)	0.0383(8)
H22	0.2094(2)	-0.2753(2)	0.2331(3)	0.051
C23	0.2112(2)	-0.3708(2)	0.1535(3)	0.0435(9)
H23	0.2274(2)	-0.3991(2)	0.2185(3)	0.058
C24	0.1975(2)	-0.4028(2)	0.0477(4)	0.0431(9)
H24	0.2054(2)	-0.4525(2)	0.0417(4)	0.057
C25	0.1721(2)	-0.3605(2)	-0.0488(3)	0.0391(9)
H25	0.1616(2)	-0.3820(2)	-0.1198(3)	0.052
C26	0.1623(2)	-0.2863(2)	-0.0398(3)	0.0351(8)
H26	0.1457(2)	-0.2582(2)	-0.1051(3)	0.047
C27	0.4002(2)	0.0270(2)	-0.2597(4)	0.0465(10)
C28	0.4296(2)	0.0987(2)	-0.1988(3)	0.0450(10)
C29	0.4569(3)	-0.0359(3)	-0.2188(5)	0.073(2)
H29a	0.479(2)	-0.0345(12)	-0.126(3)	0.109

H29b	0.503(2)	-0.0300(11)	-0.256(3)	0.109
H29c	0.4282(10)	-0.088(2)	-0.245(3)	0.109
C30	0.3743(3)	0.0289(4)	-0.3866(4)	0.082(2)
H30a	0.421(2)	0.045(2)	-0.4171(10)	0.122
H30b	0.329(2)	0.067(2)	-0.4153(10)	0.122
H30c	0.355(2)	-0.025(2)	-0.4189(11)	0.122
C31	0.5127(2)	0.1117(3)	-0.1667(4)	0.0620(13)
H31a	0.5302(6)	0.113(2)	-0.240(2)	0.093
H31b	0.5411(8)	0.0697(14)	-0.113(3)	0.093
H31c	0.5253(5)	0.162(2)	-0.124(3)	0.093
C32	0.3873(3)	0.1650(3)	-0.2642(5)	0.0691(14)
H32a	0.405(2)	0.1739(12)	-0.343(3)	0.104
H32b	0.401(2)	0.2152(14)	-0.208(2)	0.104
H32c	0.324(2)	0.1547(8)	-0.289(3)	0.104
C33	0.1174(2)	0.2831(2)	0.0852(3)	0.0308(7)
C34	0.1478(2)	0.3210(2)	0.1845(3)	0.0400(9)
H34	0.1732(2)	0.2959(2)	0.2501(3)	0.053
C35	0.1411(2)	0.3961(2)	0.1879(4)	0.0450(10)
H35	0.1625(2)	0.4209(2)	0.2555(4)	0.060
C36	0.1032(2)	0.4340(2)	0.0931(4)	0.0447(10)
H36	0.0968(2)	0.4841(2)	0.0967(4)	0.059
C37	0.0746(3)	0.3976(2)	-0.0080(4)	0.0523(11)
H37	0.0503(3)	0.4234(2)	-0.0734(4)	0.070
C38	0.0819(2)	0.3224(2)	-0.0125(3)	0.0441(9)
H38	0.0630(2)	0.2981(2)	-0.0812(3)	0.059
C39	0.2699(2)	-0.0029(2)	0.2960(3)	0.0475(10)
H39	0.2616(2)	-0.0529(2)	0.2920(3)	0.063

C40	0.3309(2)	0.0260(3)	0.2673(4)	0.0531(11)
H40	0.3635(2)	-0.0044(3)	0.2437(4)	0.071
C41	0.3427(2)	0.1007(3)	0.2742(4)	0.0578(12)
H41	0.3837(2)	0.1207(3)	0.2557(4)	0.077
C42	0.2938(3)	0.1454(2)	0.3085(4)	0.0546(11)
H42	0.3017(3)	0.1955(2)	0.3127(4)	0.073
C43	0.2335(2)	0.1161(2)	0.3364(3)	0.0493(10)
H43	0.2006(2)	0.1463(2)	0.3595(3)	0.066
C44	0.2217(2)	0.0422(2)	0.3302(3)	0.0451(9)
H44	0.1808(2)	0.0225(2)	0.3492(3)	0.060
C45	0.5294(3)	0.1630(4)	0.4982(5)	0.086(2)
H45	0.5077(3)	0.1319(4)	0.4379(5)	0.114
C46	0.6021(3)	0.1508(3)	0.5664(5)	0.079(2)
H46	0.6293(3)	0.1115(3)	0.5515(5)	0.105
C47	0.6345(3)	0.1953(3)	0.6550(5)	0.0693(14)
H47	0.6833(3)	0.1863(3)	0.7011(5)	0.092
C48	0.5947(3)	0.2535(3)	0.6758(5)	0.076(2)
H48	0.6166(3)	0.2845(3)	0.7361(5)	0.101
C49	0.5217(3)	0.2664(3)	0.6072(6)	0.080(2)
H49	0.4947(3)	0.3063(3)	0.6211(6)	0.106
C50	0.4894(3)	0.2205(3)	0.5194(5)	0.080(2)
H50	0.4402(3)	0.2286(3)	0.4740(5)	0.107
B1	0.3455(2)	0.0469(2)	-0.1201(3)	0.0320(9)

$U_{eq} = {}^1/_3[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha]$

C18	0.034(2)	0.037(2)	0.035(2)	0.000(2)	0.015(2)	0.004(2)
C19	0.029(2)	0.032(2)	0.030(2)	0.0011(14)	0.0112(14)	0.0017(14)
C20	0.029(2)	0.034(2)	0.035(2)	0.002(2)	0.014(2)	-0.001(2)
C21	0.031(2)	0.025(2)	0.036(2)	0.0016(14)	0.010(2)	-0.0003(14)
C22	0.043(2)	0.035(2)	0.035(2)	0.001(2)	0.008(2)	-0.003(2)
C23	0.049(2)	0.032(2)	0.046(2)	0.009(2)	0.009(2)	0.001(2)
C24	0.046(2)	0.025(2)	0.058(3)	-0.001(2)	0.014(2)	0.002(2)
C25	0.043(2)	0.031(2)	0.042(2)	-0.003(2)	0.010(2)	0.002(2)
C26	0.037(2)	0.031(2)	0.036(2)	0.001(2)	0.008(2)	0.001(2)
C27	0.049(2)	0.051(3)	0.050(2)	-0.012(2)	0.031(2)	-0.015(2)
C28	0.048(2)	0.050(2)	0.046(2)	-0.004(2)	0.028(2)	-0.010(2)
C29	0.076(3)	0.051(3)	0.113(5)	-0.002(3)	0.060(3)	0.004(3)
C30	0.086(4)	0.120(5)	0.049(3)	-0.024(3)	0.038(3)	-0.043(4)
C31	0.049(3)	0.069(3)	0.078(3)	-0.023(3)	0.035(2)	-0.023(2)
C32	0.082(4)	0.055(3)	0.085(4)	0.022(3)	0.048(3)	0.013(3)
C33	0.032(2)	0.026(2)	0.034(2)	0.0010(14)	0.010(2)	-0.0004(14)
C34	0.041(2)	0.038(2)	0.037(2)	-0.004(2)	0.005(2)	0.006(2)
C35	0.043(2)	0.039(2)	0.051(2)	-0.013(2)	0.012(2)	-0.001(2)
C36	0.049(2)	0.028(2)	0.061(3)	-0.003(2)	0.022(2)	-0.002(2)
C37	0.065(3)	0.034(2)	0.054(3)	0.010(2)	0.011(2)	0.005(2)
C38	0.054(2)	0.032(2)	0.041(2)	0.001(2)	0.005(2)	-0.001(2)
C39	0.052(2)	0.036(2)	0.046(2)	-0.001(2)	0.002(2)	0.002(2)
C40	0.045(2)	0.058(3)	0.054(3)	-0.003(2)	0.010(2)	0.008(2)
C41	0.042(2)	0.066(3)	0.059(3)	0.003(2)	0.005(2)	-0.010(2)
C42	0.058(3)	0.037(2)	0.057(3)	-0.001(2)	0.000(2)	-0.006(2)
C43	0.049(2)	0.049(3)	0.042(2)	-0.008(2)	0.002(2)	0.007(2)
C44	0.045(2)	0.048(2)	0.038(2)	0.002(2)	0.004(2)	0.001(2)

C45	0.085(4)	0.104(5)	0.062(3)	-0.003(3)	0.010(3)	0.027(4)
C46	0.072(4)	0.083(4)	0.074(4)	-0.007(3)	0.009(3)	0.028(3)
C47	0.058(3)	0.053(3)	0.091(4)	0.007(3)	0.013(3)	-0.001(2)
C48	0.080(4)	0.044(3)	0.109(5)	-0.007(3)	0.037(3)	-0.017(3)
C49	0.068(4)	0.049(3)	0.134(6)	0.014(3)	0.049(4)	0.008(3)
C50	0.069(3)	0.091(4)	0.081(4)	0.025(3)	0.021(3)	0.021(3)
B1	0.036(2)	0.029(2)	0.032(2)	0.002(2)	0.012(2)	0.003(2)

The form of the anisotropic displacement parameter is:
$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+ 2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$.

Table 4. Bond Distances in Compound 922, Å

Zn-N3	2.033(3)	Zn-N1	2.038(3)	Zn-N2	2.039(3)
Zn-N4	2.041(3)	O1-B1	1.363(5)	O1-C27	1.464(4)
O2-B1	1.365(5)	O2-C28	1.477(4)	N1-C4	1.374(4)
N1-C1	1.381(4)	N2-C6	1.376(4)	N2-C9	1.387(4)
N3-C11	1.376(4)	N3-C14	1.377(4)	N4-C16	1.369(4)
N4-C19	1.378(4)	C1-C20	1.383(5)	C1-C2	1.432(5)
C2-C3	1.349(5)	C3-C4	1.436(5)	C4-C5	1.407(5)
C5-C6	1.407(5)	C5-C21	1.499(5)	C6-C7	1.449(5)
C7-C8	1.351(5)	C8-C9	1.441(5)	C9-C10	1.401(5)
C10-C11	1.406(5)	C10-B1	1.575(5)	C11-C12	1.441(5)
C12-C13	1.352(5)	C13-C14	1.439(5)	C14-C15	1.402(5)
C15-C16	1.407(5)	C15-C33	1.497(5)	C16-C17	1.446(5)
C17-C18	1.346(5)	C18-C19	1.426(5)	C19-C20	1.390(5)
C21-C22	1.391(5)	C21-C26	1.395(5)	C22-C23	1.389(5)
C23-C24	1.388(6)	C24-C25	1.384(5)	C25-C26	1.384(5)
C27-C30	1.500(6)	C27-C28	1.533(5)	C27-C29	1.542(6)
C28-C31	1.491(6)	C28-C32	1.540(6)	C33-C34	1.379(5)
C33-C38	1.393(5)	C34-C35	1.387(5)	C35-C36	1.365(6)
C36-C37	1.378(6)	C37-C38	1.391(5)	C39-C44	1.374(6)
C39-C40	1.387(6)	C40-C41	1.388(6)	C41-C42	1.379(6)
C42-C43	1.375(6)	C43-C44	1.373(6)	C45-C50	1.359(8)
C45-C46	1.378(7)	C46-C47	1.355(7)	C47-C48	1.366(7)
C48-C49	1.387(8)	C49-C50	1.364(8)		

Table 5. Bond Angles in Compound 922, °

N3-Zn-N1	178.29(11)	N3-Zn-N2	90.38(11)	N1-Zn-N2	89.10(11)
N3-Zn-N4	89.70(11)	N1-Zn-N4	90.90(11)	N2-Zn-N4	177.02(11)
B1-O1-C27	107.3(3)	B1-O2-C28	106.3(3)	C4-N1-C1	106.0(3)
C4-N1-Zn	128.3(2)	C1-N1-Zn	125.5(2)	C6-N2-C9	106.5(3)
C6-N2-Zn	127.2(2)	C9-N2-Zn	126.1(2)	C11-N3-C14	106.5(3)
C11-N3-Zn	126.6(2)	C14-N3-Zn	126.5(2)	C16-N4-C19	106.3(3)
C16-N4-Zn	127.4(2)	C19-N4-Zn	126.3(2)	N1-C1-C20	124.9(3)
N1-C1-C2	109.5(3)	C20-C1-C2	125.5(3)	C3-C2-C1	107.6(3)
C2-C3-C4	107.0(3)	N1-C4-C5	124.7(3)	N1-C4-C3	109.9(3)
C5-C4-C3	125.3(3)	C6-C5-C4	124.8(3)	C6-C5-C21	116.7(3)
C4-C5-C21	118.4(3)	N2-C6-C5	125.8(3)	N2-C6-C7	109.5(3)
C5-C6-C7	124.7(3)	C8-C7-C6	107.0(3)	C7-C8-C9	107.8(3)
N2-C9-C10	126.0(3)	N2-C9-C8	109.2(3)	C10-C9-C8	124.7(3)
C9-C10-C11	124.7(3)	C9-C10-B1	117.2(3)	C11-C10-B1	117.9(3)
N3-C11-C10	125.7(3)	N3-C11-C12	109.5(3)	C10-C11-C12	124.8(3)
C13-C12-C11	107.2(3)	C12-C13-C14	107.3(3)	N3-C14-C15	126.0(3)
N3-C14-C13	109.5(3)	C15-C14-C13	124.5(3)	C14-C15-C16	124.8(3)
C14-C15-C33	117.2(3)	C16-C15-C33	118.1(3)	N4-C16-C15	125.2(3)
N4-C16-C17	109.7(3)	C15-C16-C17	125.1(3)	C18-C17-C16	106.5(3)
C17-C18-C19	108.0(3)	N4-C19-C20	124.0(3)	N4-C19-C18	109.5(3)
C20-C19-C18	126.5(3)	C1-C20-C19	128.2(3)	C22-C21-C26	118.3(3)
C22-C21-C5	122.4(3)	C26-C21-C5	119.4(3)	C23-C22-C21	121.0(4)
C24-C23-C22	119.9(4)	C25-C24-C23	119.7(4)	C26-C25-C24	120.2(4)
C25-C26-C21	120.9(3)	O1-C27-C30	109.0(3)	O1-C27-C28	102.8(3)
C30-C27-C28	116.8(4)	O1-C27-C29	105.9(3)	C30-C27-C29	109.6(4)
C28-C27-C29	112.0(4)	O2-C28-C31	109.5(3)	O2-C28-C27	102.6(3)

C31-C28-C27	117.2(4)	O2-C28-C32	105.5(3)	C31-C28-C32	109.6(4)
C27-C28-C32	111.7(4)	C34-C33-C38	118.3(3)	C34-C33-C15	121.2(3)
C38-C33-C15	120.4(3)	C33-C34-C35	120.9(4)	C36-C35-C34	120.6(4)
C35-C36-C37	119.6(4)	C36-C37-C38	120.2(4)	C37-C38-C33	120.4(4)
C44-C39-C40	120.2(4)	C39-C40-C41	119.2(4)	C42-C41-C40	120.0(4)
C43-C42-C41	120.2(4)	C44-C43-C42	120.1(4)	C43-C44-C39	120.3(4)
C50-C45-C46	119.8(6)	C47-C46-C45	121.0(5)	C46-C47-C48	119.3(5)
C47-C48-C49	120.1(6)	C50-C49-C48	119.9(5)	C45-C50-C49	119.9(6)
O1-B1-O2	113.4(3)	O1-B1-C10	120.8(3)	O2-B1-C10	125.7(3)