Inorganic Chemistry
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X-ray structure data for *r*-1-[(*Z*)-benzylideneamino-κ*N*]-*c*-3, *c*-5-diamino-κ^2*N*,*N'*-cyclohexane nitrato-κ*O* nitrato-κ^2*O*,*O'* nickel(II) ... (5)

Crystal Data

Empirical formula	$C_{13}H_{19}N_5NiO_6$	
Formula weight	400.05 g mol^{-1}	
Temperature	293(2) K	
Crystal size	0.40 x 0.30 x 0.20 mm	
Crystal description	blue block	
Space group	$P2_1/c$ (No. 14)	
Unit cell dimensions[1]	a = 8.2368(11) Å	α = 90°
	b = 10.0649(11) Å	β = 94.152(12)°
	c = 19.464(3) Å	γ =90°
Volume	1609.4(4) Å^3	
Z	4	
F(000)	832	
Denisty (calculated)	1.65 g cm^{-3}	
Denisty (measured)[2]	1.59(5) g cm^{-3}	
Linear attentuation coefficient (MoK_α)	12.49 cm^{-1}	

Data Collection

Diffractometer	Rigaku AFC6S
Radiation	MoK_α, graphite monochromated
	λ = 0.7107 Å
Scan type	ω-2θ
Scan rate	2.0° min^{-1} in ω
Scan width	(0.95 + 0.30 tan θ)°
2θ range for data collection	5.84 to 40.00°
Reflections collected	1627
Independent reflections	1493 [*R*%(int) = 4.37][3]
Transmission coefficients[4]	0.85 (min), 1.00 (max), 0.93 (average)
Data corrections	Lorentz-polarisation, absorption correction
Intensity decay of standard reflx	0.6%(3 reflxns checked every 150 data)
Index ranges	$0 \le h \le 7$, $0 \le k \le 9$, $-18 \le l \le 18$

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[1] Unit cell parameters and their esd's were determined from a least squares fitting of the setting angles of 20 reflections in the range 14.40°≤2θ≤23.70°.

[2] Determined by flotation in $CHBr_3$/hexane mixture (average of three determinations)

[3] $R(int) = \Sigma|F_o^2 - F_o^2(mean)| / \Sigma(F_o^2)$

[4] Determined from azimuthal scans.

Structure Solution

Patterson methods with SAPI91[5] and expanded using Fourier techniques with DIRDIF[6]

Refinement Method

Full-matrix least-squares on F^2 with SHELXL93[7] All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using a rigid model C_{sp2}-H = 0.93Å, $C_{sp3}(CH_3)$-H = 0.96Å, $C_{sp3}(CRH_2)$-H = 0.97Å, $C_{sp3}(CHR_2)$-H = 0.98Å, N_{sp3}-H = 0.90 Å, O_{sp3}-H = 0.82 Å with $U_{iso}[H(C_{sp2})] = 1.2U_{eq}(C_{sp2})$, $U_{iso}\{H[C_{sp3}(CH_3)\text{-}H]\} = 1.5U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CRH_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CHR_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}[H(N_{sp3}\text{-}H)] = 1.2U_{eq}(N_{sp3})$ and $U_{iso}[H(O_{sp3}\text{-}H)] = 1.5U_{eq}(O_{sp3})$

Weighting scheme	$w = [\sigma^2(F_o^2) + (0.0373P)^2 + 0.26P]^{-1}$
	$P = [\max(I_{obs},0) + 2F_c^2] / 3$
Data[8] / restraints / parameters	1491 / 0 / 226
Data to parameter ratio	6.60
Goodness-of-fit[9] on F^2	1.050
R(%)[10] indices	
$[I_o>2\sigma(I_o)]$ 1134 data	$R1 = 3.18$, $wR2 = 6.83$
All 1627 data	$R1 = 6.19$, $wR2 = 7.91$
Final difference map	
largest diff. peak[11] and hole	0.206 and -0.269 eÅ^{-3}
rms deviation from mean	0.05 eÅ^{-3}
Largest shift/ esd in final cycle	0.00

[5] F. Hai-Fu. Structure analysis programs with intelligent control, Rigaku Corporation, Tokyo, Japan, 1993.
[6] P. T. Beurskens, G. Admiraal, G. Beurskens, G. Bosman, W. P. Garcia-Granda, R. O. Gould, J. M. M. Smits and C. Smykalla. The DIRDIF program system, Technical report of the crystallography laboratory, University of Nijmegen, The Netherlands, 1992.
[7] G. M. Sheldrick, SHELXL93. Program for crystal structure refinement. Univ. of Gottingen, Germany.
[8] Two reflections suppressed in the refinement.
[9] GooF = $\{\Sigma[w(F_o^2 - F_c^2)^2] / (n\text{-}p)\}^{1/2}$ p = number of parameters, n = number of data.
[10] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$
[11] Located 1.18□ away from Ni atom.

Table 1. Fractional atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å ^{2} x 10^3) for (5). U(eq) is defined as one third of the trace of the orthogonalised Uij tensor.

Atom	x	y	z	U(eq)
Ni(1)	117(1)	1393(1)	1298(1)	32(1)
O(1)	-1960(4)	2536(4)	1503(2)	40(1)
O(2)	-1544(4)	283(4)	584(2)	42(1)
O(3)	-1304(4)	-225(4)	1663(2)	44(1)
O(4)	-2916(4)	-1382(4)	971(2)	53(1)
O(5)	-856(5)	4161(4)	2105(2)	68(1)
O(6)	-2926(5)	4540(4)	1411(2)	63(1)
N(1)	1281(5)	2765(4)	707(2)	33(1)
N(2)	1281(4)	1828(4)	2220(2)	37(1)
N(3)	1927(5)	83(4)	1081(2)	40(1)
N(4)	-1955(5)	-463(5)	1072(3)	37(1)
N(5)	-1905(6)	3759(6)	1672(2)	42(1)
C(1)	3938(6)	879(5)	1995(3)	44(2)
C(2)	3651(6)	514(6)	1242(3)	42(2)
C(3)	4003(6)	1696(6)	796(3)	44(2)
C(4)	3045(6)	2930(6)	965(3)	38(1)
C(5)	3267(6)	3269(6)	1732(3)	48(2)
C(6)	3048(6)	2104(6)	2211(3)	39(2)
C(7)	964(6)	3237(5)	102(3)	39(2)
C(8)	-557(6)	3207(5)	-341(3)	35(1)
C(9)	-437(6)	3383(5)	-1043(3)	41(2)
C(10)	-1800(8)	3393(6)	-1506(3)	51(2)
C(11)	-3307(7)	3249(5)	-1250(3)	52(2)
C(12)	-3466(7)	3097(6)	-562(3)	52(2)
C(13)	-2108(7)	3054(5)	-104(3)	43(2)

Table 2. Anisotropic displacement parameters ($Å^2 \times 10^3$) for (5). The anisotropic displacement factor exponent takes the form:
$-2\pi^2[h^2a^{*2}U11 + ... + 2hka^*b^*U12]$

Atom	U11	U22	U33	U23	U13	U12
Ni(1)	28(1)	34(1)	35(1)	0(1)	3(1)	-3(1)
O(1)	29(2)	33(3)	57(3)	-16(2)	4(2)	-1(2)
O(2)	39(2)	45(2)	42(2)	7(2)	6(2)	-5(2)
O(3)	42(2)	47(3)	43(3)	4(2)	2(2)	-13(2)
O(4)	41(2)	46(3)	72(3)	-12(2)	3(2)	-17(2)
O(5)	61(3)	54(3)	83(3)	-32(3)	-25(3)	10(2)
O(6)	46(3)	60(3)	81(3)	-4(3)	-5(2)	20(2)
N(1)	30(3)	34(3)	35(3)	1(2)	-2(2)	0(2)
N(2)	35(3)	38(3)	40(3)	2(2)	8(2)	2(2)
N(3)	34(3)	36(3)	51(3)	-3(2)	4(2)	-2(2)
N(4)	25(3)	34(3)	52(4)	-10(3)	-2(3)	1(3)
N(5)	29(3)	55(4)	41(3)	-10(3)	6(2)	2(3)
C(1)	30(3)	51(4)	53(4)	11(3)	3(3)	10(3)
C(2)	26(3)	46(4)	55(4)	-3(3)	8(3)	3(3)
C(3)	23(3)	67(5)	42(4)	-3(3)	7(3)	-10(3)
C(4)	29(3)	45(4)	38(4)	7(3)	3(3)	-13(3)
C(5)	25(3)	55(4)	62(4)	-10(3)	-1(3)	-12(3)
C(6)	30(3)	51(4)	36(3)	-3(3)	-4(3)	-5(3)
C(7)	33(4)	35(4)	48(4)	1(3)	5(3)	-9(3)
C(8)	33(4)	31(3)	40(4)	5(3)	5(3)	-2(3)
C(9)	35(4)	45(4)	45(4)	5(3)	7(3)	1(3)
C(10)	60(4)	51(4)	41(4)	15(3)	1(4)	8(3)
C(11)	39(4)	58(4)	55(5)	16(3)	-13(3)	-1(3)
C(12)	38(4)	64(5)	54(5)	11(3)	6(4)	-2(3)
C(13)	44(4)	42(4)	42(4)	6(3)	2(3)	3(3)

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

Atom	x	y	z	U(eq)
H(2A)	799(4)	2542(4)	2395(2)	45
H(2B)	1148(4)	1142(4)	2508(2)	45
H(3A)	1771(5)	-674(4)	1313(2)	48
H(3B)	1803(5)	-112(4)	629(2)	48
H(1A)	3612(6)	134(5)	2270(3)	53
H(1B)	5096(6)	1014(5)	2098(3)	53
H(2)	4381(6)	-216(6)	1139(3)	50
H(3C)	3745(6)	1463(6)	317(3)	53
H(3D)	5156(6)	1897(6)	853(3)	53
H(4)	3485(6)	3674(6)	711(3)	45
H(5A)	4349(6)	3632(6)	1830(3)	57
H(5B)	2491(6)	3955(6)	1831(3)	57
H(6)	3471(6)	2353(6)	2677(3)	47
H(7)	1829(6)	3666(5)	-86(3)	46
H(9)	586(6)	3496(5)	-1208(3)	50
H(10)	-1699(8)	3495(6)	-1975(3)	61
H(11)	-4234(7)	3255(5)	-1552(3)	62
H(12)	-4497(7)	3022(6)	-400(3)	62
H(13)	-2224(7)	2923(5)	362(3)	51

Table 4. Bond lengths(Å) and angles(°) for (5).

Nickel coordination sphere

Ni(1)-N(2)	2.021(4)	N(2)-Ni(1)-N(3)	91.0(2)
Ni(1)-N(3)	2.057(4)	N(2)-Ni(1)-N(1)	98.0(2)
Ni(1)-N(1)	2.076(4)	N(3)-Ni(1)-N(1)	86.7(2)
Ni(1)-O(1)	2.124(3)	N(2)-Ni(1)-O(1)	93.0(2)
Ni(1)-O(3)	2.155(3)	N(3)-Ni(1)-O(1)	172.6(2)
Ni(1)-O(2)	2.185(3)	N(1)-Ni(1)-O(1)	98.8(2)
		N(2)-Ni(1)-O(3)	96.0(2)
		N(3)-Ni(1)-O(3)	90.2(2)
		N(1)-Ni(1)-O(3)	165.7(2)
		O(1)-Ni(1)-O(3)	83.30(14)
		N(2)-Ni(1)-O(2)	155.6(2)
		N(3)-Ni(1)-O(2)	88.36(14)
		N(1)-Ni(1)-O(2)	106.3(2)
		O(1)-Ni(1)-O(2)	85.45(13)
		O(3)-Ni(1)-O(2)	59.57(13)

Nitrate anions

O(1)-N(5)	1.274(6)	N(5)-O(1)-Ni(1)	123.9(3)
O(2)-N(4)	1.274(5)	N(4)-O(2)-Ni(1)	90.9(3)
O(3)-N(4)	1.258(5)	N(4)-O(3)-Ni(1)	92.7(3)
O(4)-N(4)	1.224(5)	O(4)-N(4)-O(3)	121.2(5)
O(5)-N(5)	1.231(5)	O(4)-N(4)-O(2)	122.0(5)
O(6)-N(5)	1.233(5)	O(3)-N(4)-O(2)	116.8(4)
		O(5)-N(5)-O(6)	119.8(5)
		O(5)-N(5)-O(1)	120.3(5)
		O(6)-N(5)-O(1)	119.9(5)

Benzyl group

C(7)-C(8)	1.469(7)	N(1)-C(7)-C(8)	129.9(5)
C(8)-C(9)	1.388(7)	C(9)-C(8)-C(13)	118.1(5)
C(8)-C(13)	1.398(7)	C(9)-C(8)-C(7)	117.2(5)
C(9)-C(10)	1.387(7)	C(13)-C(8)-C(7)	124.7(5)
C(10)-C(11)	1.378(7)	C(10)-C(9)-C(8)	121.9(5)
C(11)-C(12)	1.364(7)	C(11)-C(10)-C(9)	118.2(5)
C(12)-C(13)	1.378(7)	C(12)-C(11)-C(10)	121.3(5)
		C(11)-C(12)-C(13)	120.4(5)
		C(12)-C(13)-C(8)	120.1(5)

Cyclohexane

N(1)-C(7)	1.278(6)	C(7)-N(1)-C(4)	113.1(4)
N(1)-C(4)	1.511(6)	C(7)-N(1)-Ni(1)	133.3(3)
N(2)-C(6)	1.483(6)	C(4)-N(1)-Ni(1)	111.1(3)
N(3)-C(2)	1.497(6)	C(6)-N(2)-Ni(1)	115.7(3)
C(1)-C(6)	1.510(7)	C(2)-N(3)-Ni(1)	117.5(3)
C(1)-C(2)	1.512(7)	C(6)-C(1)-C(2)	115.0(4)
C(2)-C(3)	1.513(7)	N(3)-C(2)-C(1)	110.7(4)
C(3)-C(4)	1.520(7)	N(3)-C(2)-C(3)	109.0(4)
C(4)-C(5)	1.530(7)	C(1)-C(2)-C(3)	110.0(5)
C(5)-C(6)	1.517(7)	C(2)-C(3)-C(4)	113.2(4)
		N(1)-C(4)-C(3)	109.9(4)
		N(1)-C(4)-C(5)	113.1(4)
		C(3)-C(4)-C(5)	111.4(4)
		C(6)-C(5)-C(4)	114.7(4)
		N(2)-C(6)-C(1)	110.3(4)
		N(2)-C(6)-C(5)	108.2(4)
		C(1)-C(6)-C(5)	112.2(4)

Selected torsion angle

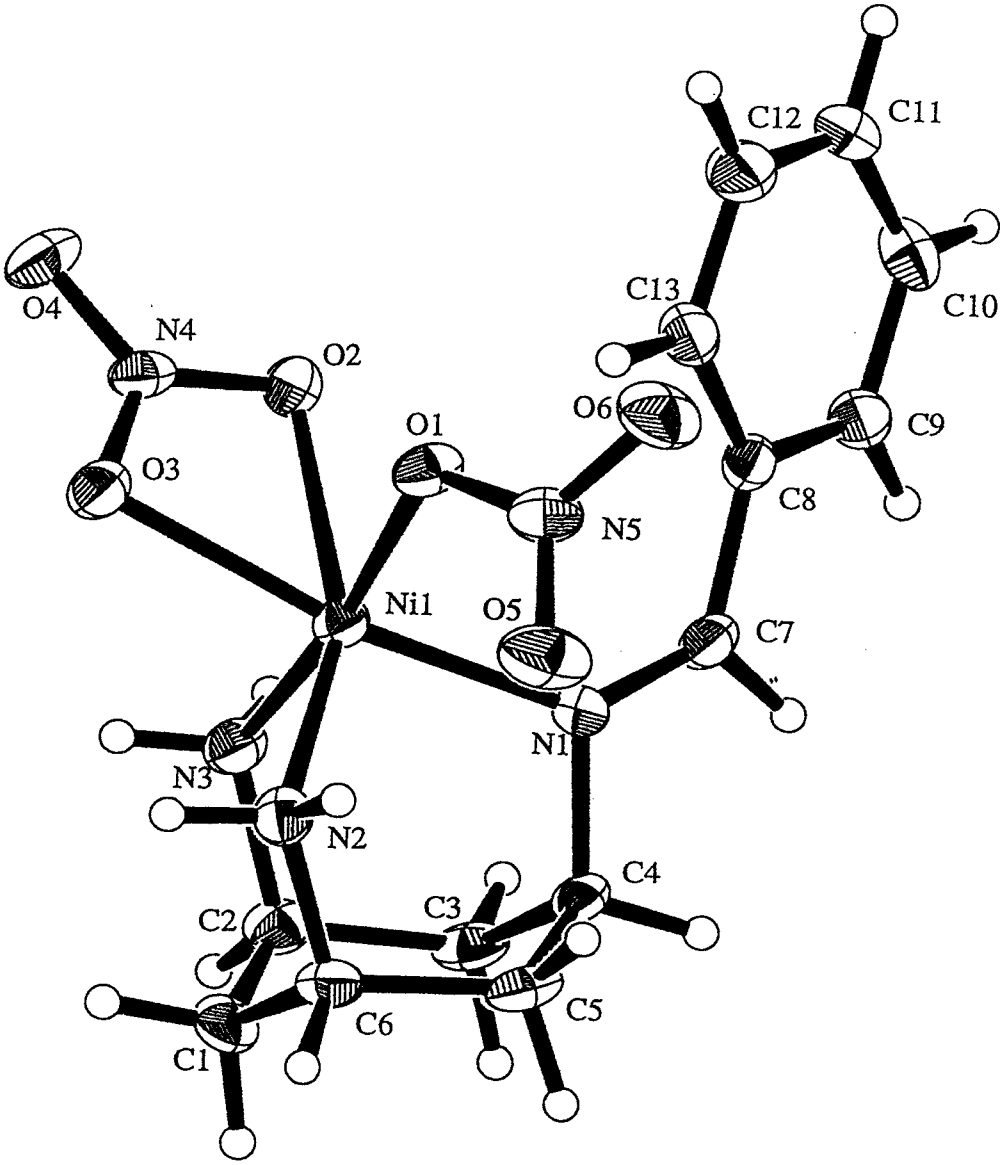
N(1)-C(7)-C(8)-C(13) 23.3(9)

Figure captions

Figure 1. ORTEP[12] plot with 30% probability thermal ellipsoids showing molecular structure of (5).

Figure 2. Stereoscopic ORTEP[12] plot with 30% probability thermal ellipsoids showing packing diagram for (5).

[12] C. K. Johnson, ORTEP, Report ORNL-5138, Oak Ridge National Laboratory, Oak Ridge, TN, 1976.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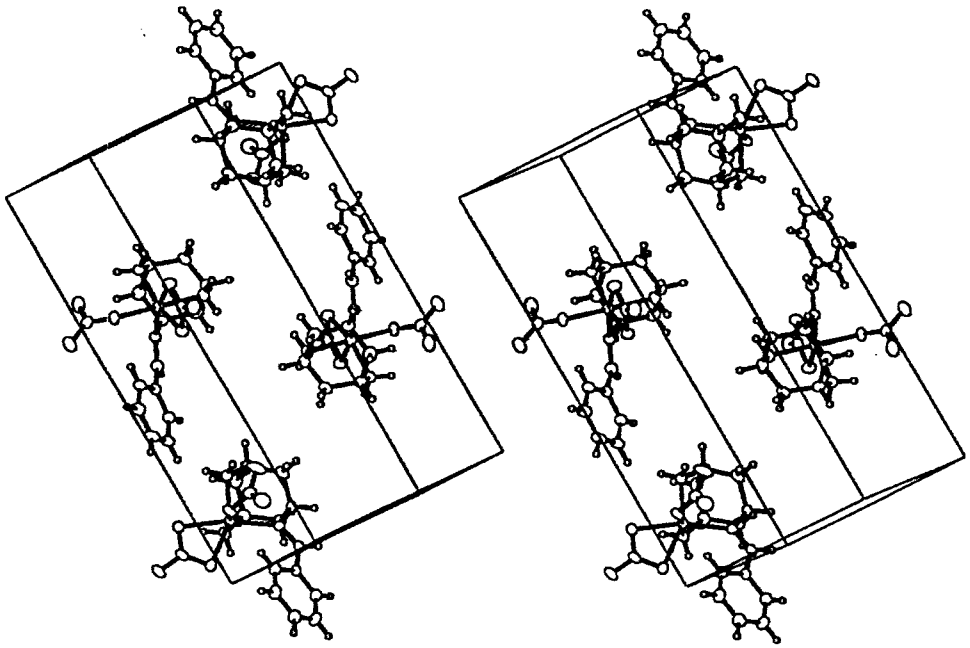
C11
C13
C10
O4
N4
O2
O6
O1
C9
O3
C8
N5
Ni1
O5
C7
N1
N3
N2
C4
C2
C3
C6
C5
C1

Fig. 2



X-ray structure for *r*-1-[(Z)-benzylideneamino-κ*N*]-*c*-3, *c*-5-diamino-κ^2*N*,*N*'-cyclohexane bis(acetato-κ*O*) copper (II) ... (6)

Crystal Data

Empirical formula	$C_{17}H_{25}CuN_3O_4$	
Formula weight	398.95 gmol^{-1}	
Temperature	293(2) K	
Crystal size	0.40 × 0.30 × 0.20 mm	
Crystal description	blue block	
Space group	Cmc21 (No. 36)	
Unit cell dimensions[1]	a = 10.275(2) Å	α = 90°
	b = 19.345(6) Å	β = 90°
	c = 9.025(4) Å	γ = 90°
Volume	1794.0(10)Å^3	
Z	4	
F(000)	836	
Denisty (calculated)	1.48 gcm^{-3}	
Denisty (measured)[2]	1.47(5) gcm^{-3}	
Linear attentuation coefficient (MoK_α)	12.43 cm^{-1}	

Data Collection

Diffractometer	Rigaku AFC6S
Radiation	MoK_α, graphite monochromated
	λ = 0.7107 Å
Scan type	ω-2θ
Scan rate	2° min^{-1} in ω
Scan width	(1.26 + 0.30 tan θ)°
2θ range for data collection	6.18 to 50.00°
Reflections collected	921
Independent reflections	894
Transmission coefficients[3]	0.70 (min), 1.00 (max), 0.94 (average)
Data corrections	Lorentz-polarisation, absorption correction[4]
Intensity decay of standard reflx	0.5% (3 reflxns checked every 150 data)
Index ranges	$0 \le h \le 12$, $0 \le k \le 20$, $0 \le l \le 10$

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[1] Unit cell parameters and their esd's were determined from a least squares fitting of the setting angles of 20 reflections in the range 14.21°≤2θ≤18.04°.
[2] Determined by flotation in $CHBr_3$/hexane mixture (average of three determinations).
[3] Determined from azimuthal scans.
[4] Empirical correction based on azimuthal scans of two reflections.

Structure Solution

Patterson methods with SAPI91[5] and expanded using Fourier techniques with DIRDIF[6]

Refinement Method

Full-matrix least-squares on F^2 with SHELXL93[7] All non-hydrogen atoms were refined anisotropically.

Hydrogen atoms were refined using a rigid model C_{sp2}-H = 0.93 Å, $C_{sp3}(CH_3)$-H = 0.96Å, $C_{sp3}(CRH_2)$-H = 0.97Å, $C_{sp3}(CHR_2)$-H = 0.98Å, N_{sp3}=0.90Å, O_{sp3}-H = 0.82 Å with $U_{iso}[H(C_{sp2})] = 1.2U_{eq}(C_{sp2})$, $U_{iso}\{H[C_{sp3}(CH_3)\text{-}H]\} = 1.5U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CRH_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CHR_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}[H(N_{sp3}\text{-}H)] = 1.2U_{eq}(N_{sp3})$ and $U_{iso}[H(O_{sp3}\text{-}H)] = 1.5U_{eq}(O_{sp3})$

Weighting scheme	$w = [\sigma^2(F_o^2) + (0.0564P)^2 + 0.18P]^{-1}$
	$P = [\max(I_{obs}, 0) + 2F_c^2] / 3$
Data / restraints / parameters	894 / 0 / 131
Data to parameter ratio	6.82
Goodness-of-fit[8] on F^2	1.073
R(%)[9] indices	
$[I_o > 2\sigma(I_o)]$ 808 data	$R1 = 3.00$, $wR2 = 7.63$
All 894 data	$R1 = 3.90$, $wR2 = 8.13$
Final difference map	
largest diff. peak[10] and hole	0.46 and -0.40 eÅ^{-3}
rms deviation from mean	0.06 eÅ^{-3}
Largest shift/ esd in final cycle	0.00

[5] F. Hai-Fu. Structure analysis programs with intelligent control, Rigaku Corporation, Tokyo, Japan, 1993.
[6] P. T. Beurskens, G. Admiraal, G. Beurskens, G. Bosman, W. P. Garcia-Granda, R. O. Gould, J. M. M. Smits and C. Smykalla. The DIRDIF program system, Technical report of the crystallography laboratory, University of Nijmegen, The Netherlands, 1992.
[7] G. M. Sheldrick, SHELXL93. Program for crystal structure refinement. Univ. of Gottingen, Germany.
[8] GooF = $\{\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)\}^{1/2}$ p = number of parameters, n = number of data.
[9] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$
[10] Located 1.04 □ away from the Cu atom.

Table 1. Fractional atomic coordinates[11] (x 10^4) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for (6). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	x	y	z	U(eq)
Cu(1)	5000	2193(1)	5000	33(1)
O(1)	3727(2)	1433(1)	5012(7)	42(1)
O(2)	2076(4)	1880(2)	3732(6)	54(1)
N(1)	5000	2682(2)	2555(8)	36(1)
N(2)	3587(4)	2864(2)	5531(6)	38(1)
C(1)	2635(4)	1393(2)	4353(7)	40(1)
C(2)	2021(5)	682(3)	4343(9)	65(2)
C(3)	5000	3880(4)	5865(10)	48(2)
C(4)	3763(5)	3603(2)	5156(8)	45(1)
C(5)	3769(6)	3705(2)	3477(7)	50(1)
C(6)	5000	3447(4)	2688(9)	44(2)
C(7)	5000	2475(3)	1229(8)	35(1)
C(8)	5000	1749(5)	703(10)	34(2)
C(9)	5000	1670(5)	-824(11)	52(3)
C(10)	5000	1000(5)	-1455(12)	59(3)
C(11)	5000	432(5)	-519(11)	50(2)
C(12)	5000	528(5)	967(11)	55(3)
C(13)	5000	1182(4)	1591(11)	44(2)

[11] Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y,z

Table 2. Anisotropic displacement parameters ($Å^2 \times 10^3$) for (6).
The anisotropic displacement factor exponent takes the form:
$-2\pi^2[h^2a^{*2}U11 + ... + 2hka^*b^*U12]$

Atom	U11	U22	U33	U23	U13	U12
Cu(1)	30(1)	36(1)	32(1)	0(1)	0	0
O(1)	31(1)	44(1)	50(2)	5(2)	-6(3)	-3(1)
O(2)	49(2)	52(2)	61(2)	-3(2)	-19(2)	7(2)
N(1)	39(3)	30(3)	38(3)	-2(2)	0	0
N(2)	35(2)	39(2)	42(2)	-1(2)	3(2)	-2(2)
C(1)	35(2)	44(2)	39(2)	-5(2)	1(2)	4(2)
C(2)	42(3)	57(3)	96(5)	-1(3)	-11(3)	-8(2)
C(3)	59(4)	44(4)	43(5)	-12(3)	0	0
C(4)	45(2)	36(2)	53(4)	-11(3)	7(3)	7(2)
C(5)	59(3)	37(2)	53(3)	0(2)	-11(3)	13(2)
C(6)	54(4)	39(3)	39(4)	5(3)	0	0
C(7)	36(3)	34(3)	35(4)	4(3)	0	0
C(8)	29(4)	42(4)	32(4)	1(3)	0	0
C(9)	81(8)	37(5)	37(6)	11(4)	0	0
C(10)	104(8)	47(5)	27(4)	-8(4)	0	0
C(11)	69(5)	41(4)	40(5)	-6(3)	0	0
C(12)	95(7)	39(4)	32(5)	1(4)	0	0
C(13)	59(5)	44(4)	30(4)	-4(3)	0	0

Table 3. Hydrogen fractional atomic coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for (6).

Atom	x	y	z	U(eq)
H(2A)	2849(4)	2719(2)	5091(6)	46
H(2B)	3459(4)	2836(2)	6516(6)	46
H(2C)	2194(40)	454(10)	5268(22)	98
H(2D)	2381(35)	414(9)	3546(34)	98
H(2E)	1097(8)	725(3)	4209(55)	98
H(3A)	5000	3759(4)	6908(10)	58
H(3B)	5000	4380(4)	5794(10)	58
H(4)	3024(5)	3860(2)	5564(8)	54
H(5A)	3666(6)	4194(2)	3268(7)	60
H(5B)	3023(6)	3467(2)	3062(7)	60
H(6)	5000	3640(4)	1684(9)	52
H(7)	5000	2815(3)	502(8)	42
H(9)	5000	2057(5)	-1435(11)	62
H(10)	5000	943(5)	-2478(12)	71
H(11)	5000	-13(5)	-909(11)	60
H(12)	5000	144(5)	1587(11)	66
H(13)	5000	1233(4)	2615(11)	53

Table 4. Bond lengths(Å) and angles(°) for (6).

Copper coordination sphere

Cu(1)-O(1)#1	1.968(3)	O(1)#1-Cu(1)-O(1)	83.3(2)
Cu(1)-O(1)	1.968(3)	O(1)#1-Cu(1)-N(2)#1	90.04(13)
Cu(1)-N(2)#1	2.006(4)	O(1)-Cu(1)-N(2)#1	164.4(3)
Cu(1)-N(2)	2.006(4)	O(1)#1-Cu(1)-N(2)	164.4(3)
Cu(1)-N(1)	2.401(7)	O(1)-Cu(1)-N(2)	90.04(13)
		N(2)#1-Cu(1)-N(2)	92.8(2)
		O(1)#1-Cu(1)-N(1)	107.4(2)
		O(1)-Cu(1)-N(1)	107.4(2)
		N(2)#1-Cu(1)-N(1)	88.0(2)
		N(2)-Cu(1)-N(1)	88.0(2)

Acetates

O(1)-C(1)	1.272(5)	C(1)-O(1)-Cu(1)	129.0(3)
O(2)-C(1)	1.238(6)	O(2)-C(1)-O(1)	125.0(4)
C(1)-C(2)	1.514(7)	O(2)-C(1)-C(2)	119.7(4)
		O(1)-C(1)-C(2)	115.3(4)

Benzyl group

N(1)-C(7)	1.262(9)	C(7)-N(1)-C(6)	113.2(6)
C(7)-C(8)	1.482(12)	C(13)-C(8)-C(9)	119.8(11)
C(8)-C(13)	1.359(13)	C(13)-C(8)-C(7)	125.1(8)
C(8)-C(9)	1.387(9)	C(9)-C(8)-C(7)	115.1(9)
C(9)-C(10)	1.414(13)	C(8)-C(9)-C(10)	120.1(10)
C(10)-C(11)	1.387(14)	C(11)-C(10)-C(9)	118.7(9)
C(11)-C(12)	1.354(13)	C(12)-C(11)-C(10)	119.6(10)
C(12)-C(13)	1.385(12)	C(11)-C(12)-C(13)	121.9(10)
		C(8)-C(13)-C(12)	119.9(9)

Cyclohexane

N(1)-C(6)	1.484(8)
N(2)-C(4)	1.480(5)
C(3)-C(4)	1.521(7)
C(3)-C(4)#1	1.521(7)
C(4)-C(5)	1.528(8)
C(5)-C(6)	1.535(7)
C(6)-C(5)#1	1.535(7)

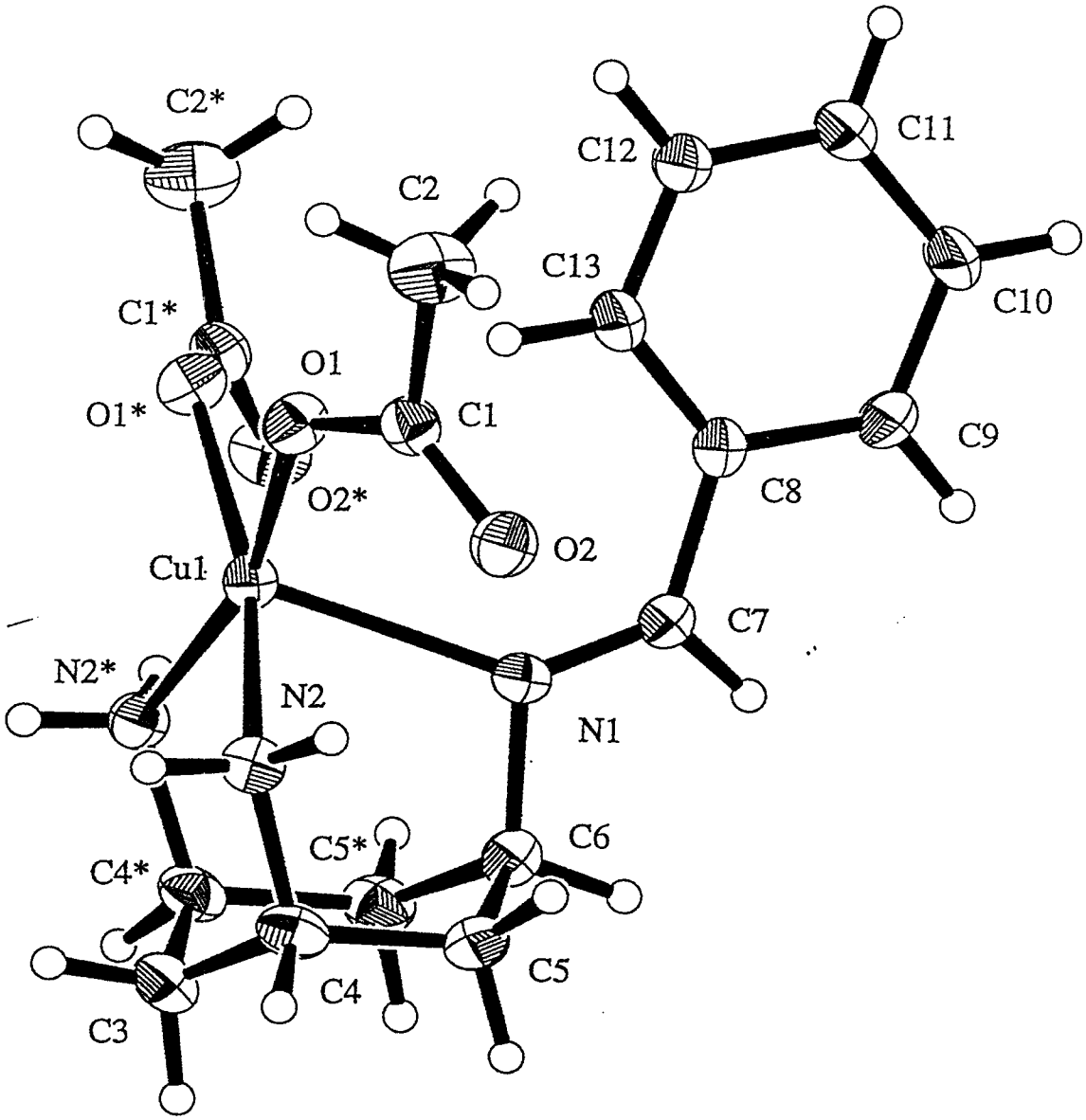
C(7)-N(1)-Cu(1)	138.3(4)
C(6)-N(1)-Cu(1)	108.5(4)
C(4)-N(2)-Cu(1)	118.8(3)
C(4)-C(3)-C(4)#1	113.4(6)
N(2)-C(4)-C(3)	110.3(5)
N(2)-C(4)-C(5)	110.7(4)
C(3)-C(4)-C(5)	111.6(5)
C(4)-C(5)-C(6)	114.9(5)
N(1)-C(6)-C(5)	111.3(4)
N(1)-C(6)-C(5)#1	111.3(4)
C(5)-C(6)-C(5)#1	111.0(6)
N(1)-C(7)-C(8)	127.2(6)

Figure captions

Figure 1. ORTEP[12] plot with 30% probability thermal ellipsoids showing molecular structure of (6).

Figure 2. Stereoscopic ORTEP[12] plot with 30% probability thermal ellipsoids showing packing diagram for (6).

[12] C. K. Johnson, ORTEP, Report ORNL-5138, Oak Ridge National Laboratory, Oak Ridge, TN, 1976.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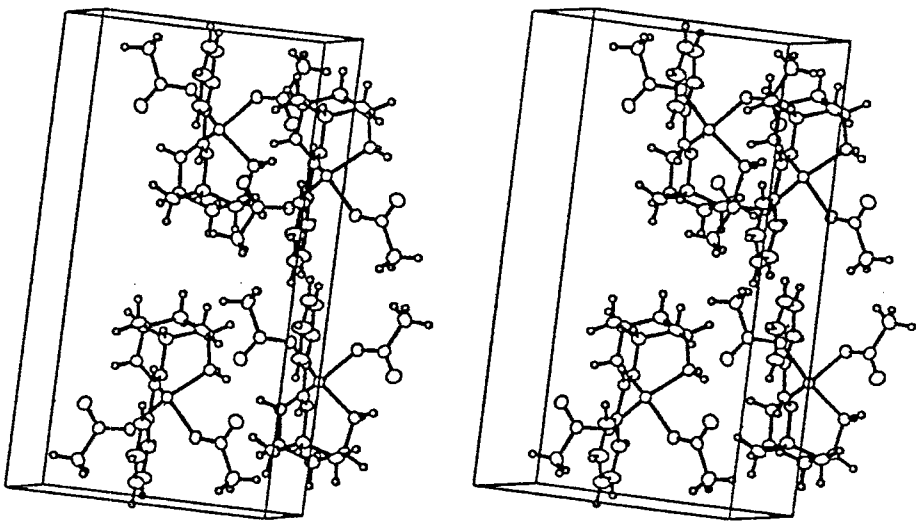
C2
C12
C11
C13
C10
C1*
O1
O1*
C1
C9
O2*
C8
O2
Cu1
C7
N2*
N2
N1
C5*
C6
C4*
C5
C4
C3

Fig 2



X-ray Structure Data for *r*-1-[(Z)-benzylideneamino-κ*N*]-*c*-3,*c*-5-diamino-κ^2*N*,*N*′-cyclohexane chloro zinc(II) tetraphenylborate methanol ... (7)

Crystal Data		
Empirical formula	$C_{38}H_{43}BClN_3OZn$	
Formula weight	669.4 gmol^{-1}	
Temperature	298(2) K	
Crystal size	0.70 x 0.70 x 0.40 mm	
Crystal description	colourless plates	
Space group	P $\bar{1}$ (No. 2)	
Unit cell dimensions[1]	a = 12.757(4) Å	α = 97.57(3)°
	b = 13.025(9) Å	β = 102.43(3)°
	c = 10.684(3) Å	γ = 87.77(4)°
Volume	1718.4(14) Å^3	
Z	2	
F(000)	704	
Density (calculated)	1.294 gcm^{-3}	
Density (measured)[2]	1.29(5) gcm^{-3}	
Linear attenuation coefficient (MoK_α)	8.26 cm^{-1}	
Data Collection		
Diffractometer	Rigaku AFC6S	
Radiation	MoK_α, graphite monochromated	
	λ = 0.71069 Å	
Scan type	ω–2θ	
Scan rate	4.0°min^{-1} in ω	
Scan width	(1.00 + 0.30tanθ)°	
2θ range for data collection	5.20 to 49.99°	
Reflections collected	4853	
Independent reflections	4558 [*R*%(int) = 2.2][3]	
Transmission coefficients[4]	0.70 (min), 1.00 (max), 0.91 (average)	
Data corrections	Lorentz-polarisation, absorption correction[5]	
Intensity decay of standard reflx	5.6% (3 reflxns checked every 150 data)	
Index ranges	$0 \le h \le 13$, $-15 \le k \le 15$, $-12 \le l \le 12$	

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[1] Unit cell parameters and their esd's were determined from a least squares fitting of the setting angles of 20 reflections in the range 13.90°≤2θ≤16.20°.

[2] Determined by flotation in $CHBr_3$/hexane mixture, average of three determinations.

[3] $R(\text{int}) = \Sigma | F_o^2 - F_o^2(\text{mean}) | \ / \ \Sigma(F_o^2)$

[4] Determined from azimuthal scans.

[5] Empirical correction based on azimuthal scans of four reflections.

Structure Solution

Patterson methods with SAPI91[6] and expanded using Fourier techniques with DIRDIF[7]

Refinement Method

Full-matrix least-squares on F^2 with SHELXL93[8] All non-hydrogen atoms were refined anisotropically.

Hydrogen atoms were refined using a rigid model C_{sp2}-H = 0.93Å, $C_{sp3}(CH_3)$-H = 0.96Å, $C_{sp3}(CRH_2)$-H = 0.97Å, $C_{sp3}(CHR_2)$-H = 0.98Å, N_{sp3}-H = 0.90Å, O_{sp3}-H = 0.82Å with $U_{iso}[H(C_{sp2})] = 1.2U_{eq}(C_{sp2})$, $U_{iso}\{H[C_{sp3}(CH_3)\text{-}H]\} = 1.5U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CRH_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CHR_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}[H(N_{sp3}\text{-}H)] = 1.2U_{eq}(N_{sp3})$ and $U_{iso}[H(O_{sp3}\text{-}H)] = 1.5U_{eq}(O_{sp3})$

Weighting scheme	$w = [\sigma^2(F_o^2) + (0.0474P)^2 + 0.81P]^{-1}$
	$P = [\max(I_{obs},0) + 2F_c^2] / 3$
Data[9] / restraints / parameters	4556 / 0 / 408
Data to parameter ratio	11.17
Goodness-of-fit[10] on F^2	1.014
R(%)[11] indices	
$[I_o>2\sigma(I_o)]$ 3338 data	$R1 = 3.97$, $wR2 = 9.15$
all 4558 data	$R1 = 7.23$, $wR2 = 10.47$
Final difference map	
largest peak[12] and hole	0.34 and -0.36 eÅ^{-3}
rms deviation from mean	0.04 eÅ^{-3}
Largest shift/esd in final cycle	0.03, U22 of O(1)

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[6] F. Hai-Fu. Structure analysis programs with intelligent control, Rigaku Corporation, Tokyo, Japan, 1993.

[7] P. T. Beurskens, G. Admiraal, G. Beurskens, G. Bosman, W. P. Garcia-Granda, R. O. Gould, J. M. M. Smits and C. Smykalla. The DIRDIF program system, Technical report of the crystallography laboratory, University of Nijmegen, The Netherlands, 1992.

[8] G. M. Sheldrick, SHELXL93. Program for crystal structure refinement. Univ. of Gottingen, Germany.

[9] Two data were supressed in the refinement.

[10] GooF = $\{\Sigma[w(F_o^2 - F_c^2)^2] / (n\text{-}p)\}^{1/2}$ p = number of parameters, n = number of data.

[11] $R1 = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$

[12] Located 0.34 Å away from O(1)

Table 1. Fractional atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for 7. U(eq) is defined as one third of the trace of the orthogonalised Uij tensor.

Atom	x	y	z	U(eq)
Zn(1)	1398(1)	1167(1)	1245(1)	54(1)
Cl(1)	1670(1)	-88(1)	-240(1)	70(1)
O(1)	4602(5)	2513(6)	5753(7)	209(3)
N(1)	2380(3)	2255(2)	2417(3)	57(1)
N(2)	128(3)	2111(2)	692(3)	57(1)
N(3)	809(3)	659(2)	2687(3)	61(1)
C(1)	1771(3)	2899(3)	3295(4)	63(1)
C(2)	841(3)	3458(3)	2495(4)	66(1)
C(3)	-122(3)	2795(3)	1831(4)	60(1)
C(4)	-465(3)	2149(3)	2760(4)	67(1)
C(5)	440(4)	1552(3)	3537(4)	65(1)
C(6)	1377(4)	2246(3)	4184(4)	69(1)
C(7)	3385(4)	2425(3)	2612(4)	70(1)
C(8)	4122(3)	1986(3)	1810(4)	62(1)
C(9)	5206(4)	1914(4)	2355(5)	91(2)
C(10)	5925(4)	1546(5)	1603(7)	111(2)
C(11)	5586(5)	1269(5)	320(7)	105(2)
C(12)	4510(4)	1334(4)	-245(5)	91(2)
C(13)	3788(4)	1694(3)	506(5)	73(1)
C(14)	7414(3)	3662(3)	8016(3)	48(1)
C(15)	7480(3)	3281(3)	9191(4)	63(1)
C(16)	6980(3)	3751(4)	10154(4)	75(1)
C(17)	6407(3)	4651(4)	9988(5)	75(1)
C(18)	6348(3)	5075(3)	8866(5)	72(1)
C(19)	6844(3)	4589(3)	7918(4)	60(1)
C(20)	9315(3)	2996(3)	7537(3)	46(1)
C(21)	10002(3)	2185(3)	7239(4)	58(1)
C(22)	11120(4)	2229(4)	7698(4)	72(1)
C(23)	11574(4)	3095(4)	8461(4)	70(1)
C(24)	10938(3)	3903(3)	8802(4)	64(1)
C(25)	9826(3)	3846(3)	8351(4)	54(1)
C(26)	7836(3)	3711(3)	5655(3)	49(1)
C(27)	8649(3)	4167(3)	5253(3)	51(1)
C(28)	8458(4)	4740(3)	4210(4)	62(1)
C(29)	7452(4)	4867(3)	3521(4)	76(1)
C(30)	6615(4)	4426(4)	3879(5)	97(2)
C(31)	6814(4)	3862(4)	4915(4)	81(1)
C(32)	7517(3)	1893(3)	6357(4)	55(1)
C(33)	7687(4)	1385(3)	5192(4)	80(1)
C(34)	7260(5)	412(4)	4650(5)	100(2)
C(35)	6627(5)	-63(4)	5300(8)	109(2)
C(36)	6462(4)	398(4)	6448(7)	104(2)

Atom	x	y	z	U(eq)
C(37)	6894(3)	1348(3)	6965(5)	74(1)
C(38)	4315(7)	2812(7)	6884(8)	169(3)
B(1)	8015(3)	3056(3)	6903(4)	48(1)

Table 2. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **7**. The anisotropic displacement factor exponent takes the form:
$-2\pi^2[h^2a^{*2}U11 + ... + 2hka^*b^*U12]$

Atom	U11	U22	U33	U23	U13	U12
Zn(1)	57(1)	49(1)	57(1)	0(1)	18(1)	3(1)
Cl(1)	68(1)	62(1)	77(1)	-14(1)	26(1)	2(1)
O(1)	146(5)	280(9)	205(7)	90(6)	-2(5)	-58(5)
N(1)	55(2)	49(2)	65(2)	3(2)	12(2)	0(2)
N(2)	70(2)	54(2)	46(2)	6(2)	12(2)	7(2)
N(3)	71(2)	52(2)	61(2)	11(2)	16(2)	4(2)
C(1)	69(3)	51(2)	62(3)	-6(2)	8(2)	-3(2)
C(2)	87(3)	46(2)	67(3)	0(2)	24(2)	11(2)
C(3)	64(3)	55(2)	58(3)	4(2)	10(2)	19(2)
C(4)	70(3)	68(3)	63(3)	-8(2)	28(2)	3(2)
C(5)	84(3)	64(3)	52(2)	10(2)	27(2)	2(2)
C(6)	85(3)	66(3)	51(3)	2(2)	10(2)	9(2)
C(7)	78(3)	56(3)	71(3)	9(2)	6(3)	-4(2)
C(8)	55(3)	58(3)	76(3)	16(2)	12(2)	-5(2)
C(9)	59(3)	116(4)	95(4)	22(3)	2(3)	-10(3)
C(10)	58(3)	147(6)	135(6)	46(5)	19(4)	8(3)
C(11)	84(5)	117(5)	133(5)	44(4)	51(4)	16(3)
C(12)	82(4)	109(4)	91(4)	25(3)	33(3)	2(3)
C(13)	56(3)	81(3)	87(4)	24(3)	17(3)	-1(2)
C(14)	41(2)	53(2)	49(2)	9(2)	4(2)	-7(2)
C(15)	58(3)	77(3)	55(3)	17(2)	13(2)	12(2)
C(16)	63(3)	115(4)	51(3)	18(3)	17(2)	1(3)
C(17)	50(3)	103(4)	67(3)	-10(3)	14(2)	2(3)
C(18)	58(3)	66(3)	87(3)	0(3)	12(2)	5(2)
C(19)	61(3)	59(3)	59(3)	9(2)	13(2)	-1(2)
C(20)	49(2)	51(2)	41(2)	14(2)	12(2)	0(2)
C(21)	64(3)	55(2)	58(2)	12(2)	18(2)	3(2)
C(22)	60(3)	84(3)	79(3)	27(3)	27(2)	25(3)
C(23)	52(3)	93(4)	70(3)	30(3)	9(2)	-4(3)
C(24)	61(3)	72(3)	58(3)	16(2)	6(2)	-12(2)
C(25)	50(3)	52(2)	57(2)	9(2)	6(2)	-2(2)
C(26)	57(3)	46(2)	44(2)	4(2)	7(2)	-7(2)
C(27)	62(3)	47(2)	45(2)	5(2)	16(2)	0(2)

Atom	x	y	z	U(eq)
H(22)	11552(4)	1670(4)	7484(4)	86
H(23)	12315(4)	3132(4)	8746(4)	84
H(24)	11242(3)	4489(3)	9330(4)	76
H(25)	9404(3)	4399(3)	8605(4)	64
H(27)	9352(3)	4088(3)	5698(3)	61
H(28)	9032(4)	5038(3)	3986(4)	74
H(29)	7327(4)	5243(3)	2820(4)	92
H(30)	5916(4)	4509(4)	3423(5)	116
H(31)	6234(4)	3568(4)	5130(4)	97
H(33)	8106(4)	1707(3)	4748(4)	96
H(34)	7400(5)	93(4)	3870(5)	120
H(35)	6316(5)	-699(4)	4946(8)	131
H(36)	6051(4)	69(4)	6895(7)	125
H(37)	6764(3)	1644(3)	7760(5)	89
H(38A)	4454(49)	2258(19)	7409(26)	253
H(38B)	3563(13)	2984(49)	6728(9)	253
H(38C)	4721(37)	3408(31)	7323(29)	253

Table 4. Bond lengths(Å) and angles(°) for 7.

Zinc coordination sphere

Zn(1)-N(1)	2.035(3)	N(1)-Zn(1)-N(3)	95.72(13)
Zn(1)-N(2)	2.026(3)	N(1)-Zn(1)-Cl(1)	132.68(10)
Zn(1)-N(3)	2.045(3)	N(2)-Zn(1)-N(1)	97.72(13)
Zn(1)-Cl(1)	2.195(2)	N(2)-Zn(1)-N(3)	94.97(13)
		N(2)-Zn(1)-Cl(1)	115.20(9)
		N(3)-Zn(1)-Cl(1)	113.12(10)

Benzyl group

N(1)-C(7)	1.277(5)	N(1)-C(7)-C(8)	126.8(4)
C(7)-C(8)	1.456(6)	C(13)-C(8)-C(9)	118.3(4)
C(8)-C(13)	1.373(6)	C(13)-C(8)-C(7)	122.1(4)
C(8)-C(9)	1.384(6)	C(9)-C(8)-C(7)	119.5(5)
C(9)-C(10)	1.377(7)	C(10)-C(9)-C(8)	120.5(5)
C(10)-C(11)	1.350(8)	C(11)-C(10)-C(9)	120.5(5)
C(11)-C(12)	1.379(7)	C(10)-C(11)-C(12)	120.1(5)
C(12)-C(13)	1.377(6)	C(13)-C(12)-C(11)	119.5(5)
		C(8)-C(13)-C(12)	121.1(5)

Cyclohexane

N(1)-C(1)	1.497(5)	C(7)-N(1)-C(1)	116.5(3)
N(2)-C(3)	1.495(4)	C(7)-N(1)-Zn(1)	133.4(3)
N(3)-C(5)	1.503(5)	C(1)-N(1)-Zn(1)	109.8(2)
C(1)-C(2)	1.525(5)	C(3)-N(2)-Zn(1)	110.9(2)
C(1)-C(6)	1.527(6)	C(5)-N(3)-Zn(1)	111.1(2)
C(2)-C(3)	1.518(6)	N(1)-C(1)-C(2)	109.8(3)
C(3)-C(4)	1.523(5)	N(1)-C(1)-C(6)	111.5(3)
C(4)-C(5)	1.521(6)	C(2)-C(1)-C(6)	111.3(4)
C(5)-C(6)	1.513(6)	C(3)-C(2)-C(1)	115.7(3)
		N(2)-C(3)-C(2)	110.0(3)
		N(2)-C(3)-C(4)	110.4(3)
		C(2)-C(3)-C(4)	111.4(3)
		C(5)-C(4)-C(3)	114.9(3)
		N(3)-C(5)-C(6)	110.0(3)
		N(3)-C(5)-C(4)	110.5(3)
		C(6)-C(5)-C(4)	111.4(3)
		C(5)-C(6)-C(1)	115.0(3)

Tetraphenylborate anion

C(14)-C(19)	1.388(5)
C(14)-C(15)	1.394(5)
C(14)-B(1)	1.651(6)
C(15)-C(16)	1.388(6)
C(16)-C(17)	1.367(6)
C(17)-C(18)	1.372(6)
C(18)-C(19)	1.379(6)
C(20)-C(21)	1.395(5)
C(20)-C(25)	1.402(5)
C(20)-B(1)	1.654(5)
C(21)-C(22)	1.406(6)
C(22)-C(23)	1.370(6)
C(23)-C(24)	1.363(6)
C(24)-C(25)	1.398(5)
C(26)-C(27)	1.385(5)
C(26)-C(31)	1.393(5)
C(26)-B(1)	1.646(5)
C(27)-C(28)	1.395(5)
C(28)-C(29)	1.352(6)
C(29)-C(30)	1.377(6)
C(30)-C(31)	1.381(6)
C(32)-C(33)	1.386(6)
C(32)-C(37)	1.395(5)
C(32)-B(1)	1.649(6)
C(33)-C(34)	1.401(7)
C(34)-C(35)	1.382(8)
C(35)-C(36)	1.349(8)
C(36)-C(37)	1.372(7)

C(19)-C(14)-C(15)	113.8(4)
C(19)-C(14)-B(1)	125.1(3)
C(15)-C(14)-B(1)	121.0(3)
C(16)-C(15)-C(14)	123.5(4)
C(17)-C(16)-C(15)	120.0(4)
C(16)-C(17)-C(18)	118.7(4)
C(17)-C(18)-C(19)	120.3(4)
C(18)-C(19)-C(14)	123.7(4)
C(21)-C(20)-C(25)	114.7(3)
C(21)-C(20)-B(1)	125.1(3)
C(25)-C(20)-B(1)	120.0(3)
C(20)-C(21)-C(22)	122.5(4)
C(23)-C(22)-C(21)	120.0(4)
C(24)-C(23)-C(22)	120.0(4)
C(23)-C(24)-C(25)	119.5(4)
C(24)-C(25)-C(20)	123.4(4)
C(27)-C(26)-C(31)	114.1(4)
C(27)-C(26)-B(1)	124.8(3)
C(31)-C(26)-B(1)	121.1(3)
C(26)-C(27)-C(28)	122.9(4)
C(29)-C(28)-C(27)	120.8(4)
C(28)-C(29)-C(30)	118.5(4)
C(29)-C(30)-C(31)	120.1(5)
C(30)-C(31)-C(26)	123.5(4)
C(33)-C(32)-C(37)	114.5(4)
C(33)-C(32)-B(1)	120.2(4)
C(37)-C(32)-B(1)	125.3(4)
C(32)-C(33)-C(34)	123.3(5)
C(35)-C(34)-C(33)	118.5(5)
C(36)-C(35)-C(34)	119.8(6)
C(35)-C(36)-C(37)	120.6(6)
C(36)-C(37)-C(32)	123.2(5)
C(26)-B(1)-C(32)	106.9(3)
C(26)-B(1)-C(14)	109.9(3)
C(32)-B(1)-C(14)	111.8(3)
C(26)-B(1)-C(20)	108.7(3)
C(32)-B(1)-C(20)	111.4(3)
C(14)-B(1)-C(20)	108.0(3)

Methanol

O(1)-C(38)	1.342(8)

Selected torsion angles

N(1)-C(7)-C(8)-C(13)	28.3(7)
Zn(1)-N(1)-C(7)-C(8)	14.4(7)

Figure captions

Figure 1. ORTEP[13] plot with 30% probability thermal ellipsoids showing molecular structure of the *r*-1-[(Z)-benzylideneamino-κ*N*]-*c*-3, *c*-5-diamino-κ^2*N*,*N*′-cyclohexane chloro zinc(II) cation.

Figure 2. Stereoscopic ORTEP[13] plot with 30% probability thermal ellipsoids showing packing diagram for **7**.

[13] C. K. Johnson, ORTEP, Report ORNL-5138, Oak Ridge National Laboratory, Oak Ridge, TN, 1976.

Fig 1

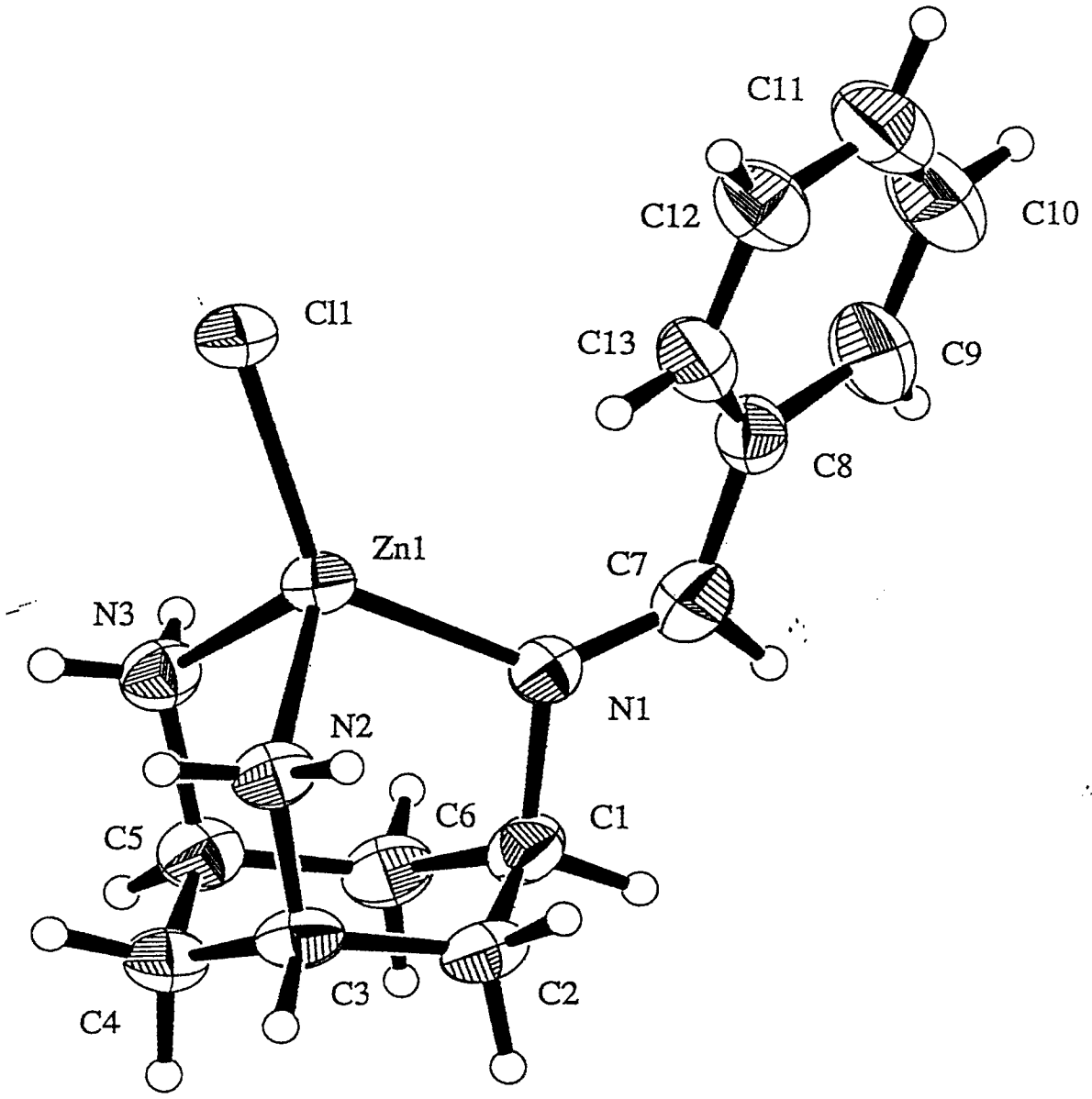
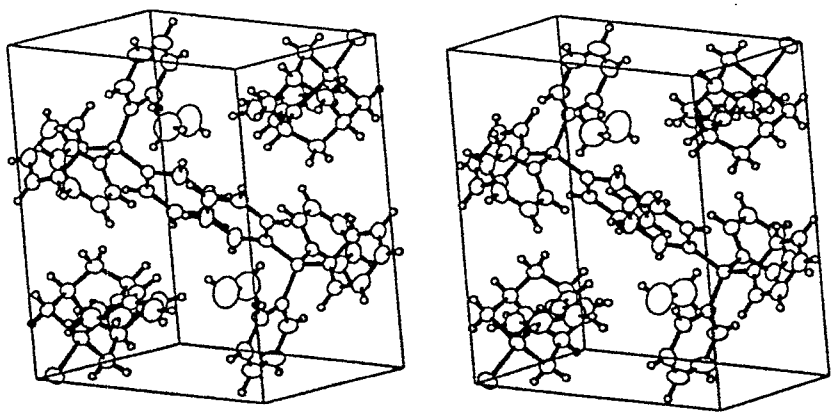


Fig 2



X-ray structure for *r*-1-[(*Z*)-3-hydroxybenzylideneamino-κ*N*]-*c*-3,*c*-5-diamino-κ²*N*,*N*'-cyclohexane bis(acetato-κ*O*) copper (II) ... (8)

Crystal Data

Empirical formula	$C_{17} H_{25} Cu N_3 O_5$
Formula weight	414.95 $gmol^{-1}$
Temperature	294(2) K
Crystal size	0.50 × 0.30 × 0.20 mm
Crystal description	blue block
Space group	Pnma (No. 62)
Unit cell dimensions[1]	a = 20.183(11) Å α = 90°
	b = 10.336(8) Å β = 90°
	c = 8.737(3) Å γ = 90°
Volume	1823(2) $Å^3$
Z	4
F(000)	868
Denisty (calculated)	1.51 g cm^{-3}
Denisty (measured)[2]	1.50(5) g cm^{-3}
Linear attentuation coefficient (MoK_α)	12.31 cm^{-1}

Data Collection

Diffractometer	Rigaku AFC6S
Radiation	MoK_α, graphite monochromated
	λ = 0.7107 Å
Scan type	ω-2θ
Scan rate	1.0° min^{-1} in ω
Scan width	(1.16 + 0.30 tan θ)°
2θ range for data collection	5.08 to 50.00°
Reflections collected	1648
Independent reflections	1494
Transmission coefficients[3]	0.80 (min), 1.00(max), 0.95(average)
Data corrections	Lorentz-polarisation
Intensity decay of standard reflx	0.3% (3 reflxns checked every 150 data)
Index ranges	$0 \le h \le 24$, $0 \le k \le 11$, $0 \le l \le 10$

[1] Unit cell parameters and their esd's were determined from a least squares fitting of the setting angles of 20 reflections in the range 14.67°≤2θ≤18.05°.
[2] Determined by flotation in $CHBr_3$/hexane mixture (average of three determinations)
[3] Determined from azimuthal scans.

Structure Solution

Patterson methods with SAPI91[4] and expanded using Fourier techniques with DIRDIF[5]

Refinement Method

Full-matrix least-squares on F^2 with SHELXL93[6] All non-hydrogen atoms were refined anisotropically.

Hydrogen atoms were refined using a rigid model C_{sp2}-H = 0.93Å, $C_{sp3}(CH_3)$-H = 0.96Å, $C_{sp3}(CRH_2)$-H = 0.97Å, $C_{sp3}(CHR_2)$-H = 0.98Å, N_{sp3}-H = 0.90Å, O_{sp3}-H = 0.82Å with $U_{iso}[H(C_{sp2})] = 1.2U_{eq}(C_{sp2})$, $U_{iso}\{H[C_{sp3}(CH_3)\text{-}H]\} = 1.5U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CRH_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CHR_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}[H(N_{sp3}\text{-}H)] = 1.2U_{eq}(N_{sp3})$ and $U_{iso}[H(O_{sp3}\text{-}H)] = 1.5U_{eq}(O_{sp3})$

Weighting scheme	$w = [\sigma^2(F_o^2) + (0.0472P)^2 + 1.65P]^{-1}$
	$P = [\max(I_{obs}, 0) + 2F_c^2] / 3$
Data / restraints / parameters	1494 / 0 / 148
Data to parameter ratio	10.09
Goodness-of-fit[7] on F^2	1.01
R(%)[8] indices	
$[I_o > 2\sigma(I_o)]$ 1149 data	$R1 = 3.40$, $wR2 = 8.08$
All 1494 data	$R1 = 5.88$, $wR2 = 9.29$
Final difference map	
largest diff. peak[9] and hole	0.32 and -0.27 eÅ^{-3}
rms deviation from mean	0.06 eÅ^{-3}
Largest shift/ esd in final cycle	0.00

[4] F. Hai-Fu. Structure analysis programs with intelligent control, Rigaku Corporation, Tokyo, Japan, 1993.
[5] P. T. Beurskens, G. Admiraal, G. Beurskens, G. Bosman, W. P. Garcia-Granda, R. O. Gould, J. M. M. Smits and C. Smykalla. The DIRDIF program system, Technical report of the crystallography laboratory, University of Nijmegen, The Netherlands, 1992.
[6] G. M. Sheldrick, SHELXL93. Program for crystal structure refinement. Univ. of Gottingen, Germany.
[7] GooF = $\{\Sigma[w(F_o^2 - F_c^2)^2] / (n\text{-}p)\}^{1/2}$ p = number of parameters, n = number of data.
[8] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$
[9] Located 1.04Å away from the Cu atom.

Table 1. Fractional atomic coordinates[10] (x 10^4) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for (8). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	x	y	z	U(eq)
Cu(1)	2358(1)	2500	2773(1)	30(1)
O(2)	1620(1)	1247(2)	2597(2)	38(1)
O(3)	1973(1)	-358(3)	4093(3)	50(1)
O(4)	678(2)	2500	10553(4)	95(2)
N(1)	2714(2)	2500	5387(4)	30(1)
N(2)	3005(1)	1090(3)	2358(3)	36(1)
C(1)	3445(2)	2500	5373(6)	34(1)
C(2)	3728(2)	3716(4)	4625(4)	39(1)
C(3)	3701(2)	1268(4)	2876(4)	38(1)
C(4)	3992(3)	2500	2204(6)	44(1)
C(5)	2453(2)	2500	6726(6)	32(1)
C(6)	1743(2)	2500	7099(5)	29(1)
C(7)	1250(2)	2500	5997(6)	36(1)
C(8)	594(3)	2500	6435(6)	41(1)
C(9)	413(3)	2500	7962(5)	38(1)
C(10)	897(3)	2500	9079(5)	47(2)
C(11)	1561(3)	2500	8645(6)	45(2)
C(12)	1534(2)	229(4)	3403(4)	38(1)
C(13)	823(2)	-236(4)	3504(5)	56(1

[10] Symmetry transformations used to generate equivalent atoms:
#1 x,-y + 1/2,z

Table 2. Anisotropic displacement parameters ($Å^2 \times 10^3$) for (8).

The anisotropic displacement factor exponent takes the form:

$-2\pi^2[h^{4*2}U11 + ... + 2hka^*b^*U12]$

Atom	U11	U22	U33	U23	U13	U12
Cu(1)	34(1)	32(1)	24(1)	0	-1(1)	0
O(2)	43(1)	36(1)	35(1)	2(1)	-7(1)	-6(1)
O(3)	63(2)	45(2)	42(2)	11(1)	5(1)	3(2)
O(4)	38(2)	224(7)‡	23(2)	0	3(2)	0
N(1)	32(2)	32(2)	25(2)	0	-1(2)	0
N(2)	44(2)	37(2)	29(2)	-4(1)	-1(1)	1(1)
C(1)	29(3)	48(3)	24(2)	0	-5(2)	0
C(2)	36(2)	48(2)	32(2)	-3(2)	-1(2)	-4(2)
C(3)	36(2)	41(2)	35(2)	-3(2)	3(2)	10(2)
C(4)	39(3)	61(4)	32(3)	0	10(2)	0
C(5)	39(3)	30(3)	26(2)	0	-4(2)	0
C(6)	35(3)	25(2)	25(2)	0	0(2)	0
C(7)	43(3)	40(3)	24(3)	0	0(2)	0
C(8)	38(3)	58(4)	26(3)	0	-8(2)	0
C(9)	32(3)	51(3)	31(3)	0	-2(2)	0
C(10)	40(3)	81(4)	21(3)	0	0(2)	0
C(11)	42(3)	70(4)	24(3)	0	-3(2)	0
C(12)	51(2)	34(2)	28(2)	-8(2)	3(2)	-2(2)
C(13)	59(3)	52(3)	57(3)	0(2)	7(2)	-19(2)

‡ The thermal ellipsoid of O(4) is elongated along the axis perpendicular to the mirror plane. We have interpreted this as thermal motion/disorder in the crystal due to the hydrogen bond contacts that can be made between O(4) and both O(2), O(2*) in adjacent molecules. The disorder orginates from oxygen O(4) and its asociated hydrogen disordered between *intermolecular* hydrogen bond formation with one acetate and hydrogen bond formation with the other acetate. The oxygen (O4) and its hydrogen have been placed on the mirror plane in the refinement presented here, however we also tried a model which had O(4) at half occupancy either side of the mirror plane; this resulted in worse R factors for the structure.

Table 3. Hydrogen fractional atomic coordinates (× 10^4) and isotropic displacement parameters ($Å^2$ × 10^3) for (8).

Atom	x	y	z	U(eq)
H(1)	3584(8)	2500	6387(58)	41
H(4)	2867(4)	449(20)	2743(11)	43
H(2B)	3011(1)	970(5)	1461(27)	43
H(2C)	4190(11)	3811(5)	4944(9)	46
H(2D)	3484(6)	4468(19)	5011(10)	46
H(3)	3955(10)	556(29)	2521(15)	45
H(4)	961(36)	2500	11077(66)	143
H(4A)	4475(18)	2500	2377(8)	53
H(4B)	3916(3)	2500	1087(42)	53
H(5)	2754(16)	2500	7578(45)	38
H(7)	1366(7)	2500	4928(58)	43
H(8)	261(19)	2500	5676(44)	49
H(9)	-22(25)	2500	8228(16)	45
H(11)	1870(20)	2500	9351(47)	54
H(13A)	579(4)	75(23)	2636(18)	84
H(13B)	625(5)	86(23)	4427(17)	84
H(13C)	815(2)	-1165(4)	3514(33)	84

Table 4. Bond lengths(Å) and angles(°) for (8).

Copper coordination sphere

Cu(1)-O(2)	1.980(2)	O(2)-Cu(1)-O(2)#1	81.8(2)
Cu(1)-O(2)#1	1.980(2)	O(2)-Cu(1)-N(2)#1	163.46(10)
Cu(1)-N(2)#1	1.990(3)	O(2)#1-Cu(1)-N(2)#1	89.99(12)
Cu(1)-N(2)	1.990(3)	O(2)-Cu(1)-N(2)	89.99(12)
Cu(1)-N(1)	2.395(4)	O(2)#1-Cu(1)-N(2)	163.46(10)
		N(2)#1-Cu(1)-N(2)	94.2(2)
		O(2)-Cu(1)-N(1)	107.43(9)
		O(2)#1-Cu(1)-N(1)	107.43(9)
		N(2)#1-Cu(1)-N(1)	88.68(10)
		N(2)-Cu(1)-N(1)	88.68(10)

Acetates

O(2)-C(12)	1.278(4)	C(12)-O(2)-Cu(1)	126.8(2)
O(3)-C(12)	1.232(4)	O(3)-C(12)-O(2)	125.2(3)
C(12)-C(13)	1.515(5)	O(3)-C(12)-C(13)	119.8(4)
		O(2)-C(12)-C(13)	115.0(3)

Benzyl group

N(1)-C(5)	1.283(6)	C(7)-C(6)-C(11)	118.9(5)
O(4)-C(10)	1.362(6)	C(7)-C(6)-C(5)	123.1(4)
C(6)-C(7)	1.384(7)	C(11)-C(6)-C(5)	118.0(4)
C(6)-C(11)	1.399(6)	N(1)-C(5)-C(6)	127.1(4)
C(6)-C(5)	1.470(6)	C(8)-C(7)-C(6)	119.8(5)
C(7)-C(8)	1.378(7)	C(7)-C(8)-C(9)	121.4(5)
C(8)-C(9)	1.383(7)	C(10)-C(11)-C(6)	121.0(5)
C(11)-C(10)	1.392(7)	O(4)-C(10)-C(9)	116.0(5)
C(10)-C(9)	1.382(7)	O(4)-C(10)-C(11)	124.8(5)
		C(10)-C(9)-C(8)	119.6(5)
		C(9)-C(10)-C(11)	119.2(5)

Cyclohexane

N(1)-C(1)	1.475(6)	C(5)-N(1)-C(1)	114.8(4)
N(2)-C(3)	1.487(4)	C(5)-N(1)-Cu(1)	138.3(3)
C(1)-C(2)#1	1.527(5)	C(1)-N(1)-Cu(1)	106.9(3)
C(1)-C(2)	1.527(5)	C(3)-N(2)-Cu(1)	118.2(2)
C(3)-C(4)	1.520(5)	N(1)-C(1)-C(2)#1	112.2(3)
C(3)-C(2)#1	1.529(5)	N(1)-C(1)-C(2)	112.2(3)
C(2)-C(3)#1	1.529(5)	C(2)#1-C(1)-C(2)	110.8(4)
C(4)-C(3)#1	1.520(5)	N(2)-C(3)-C(4)	110.6(3)
		N(2)-C(3)-C(2)#1	109.8(3)
		C(4)-C(3)-C(2)#1	111.3(4)
		C(1)-C(2)-C(3)#1	115.0(3)
		C(3)#1-C(4)-C(3)	113.8(4)
		C(10)-C(9)-C(8)	119.6(5)

Selected torsion angles

N1 - C5 - C6 - C11 180

Hydrogen bond contacts

O(4)....O(2)	2.882(3) *(Intermolecular)*	O(4)-H(4)-(O2)	139.5(4)
N(2)....O(3)	2.454(2) *(Intramolecular)*	N(2)-H(2B)-(O3)	140.0(4)

Figure captions

Figure 1. ORTEP[11] plot with 30% probability thermal ellipsoids showing molecular structure of (8).

Figure 2. Stereoscopic ORTEP[11] plot with 30% probability thermal ellipsoids showing packing diagram for (8).

[11] C. K. Johnson, ORTEP, Report ORNL-5138, Oak Ridge National Laboratory, Oak Ridge, TN, 1976.

X-ray structure for *r*-1-[(*Z*)-3,5-dimethoxybenzylideneamino-κ*N*]-*c*-3,*c*-5-diamino-κ^2*N*,*N*'-cyclohexane dichlorocopper (II) ... (q)

Crystal Data

Empirical formula	$C_{15}H_{23}Cl_2CuN_3O_2$	
Formula weight	411.80 gmol^{-1}	
Temperature	296(2) K	
Crystal size	0.30 × 0.20 × 0.20 mm	
Crystal description	green block	
Space group	$P\bar{1}$ (No.2)	
Unit cell dimensions[1]	a = 9.873(7) Å	α = 107.44(4)°
	b = 11.348(5) Å	β = 114.62(4)°
	c = 9.343(4) Å	γ = 73.35(4)°
Volume	891.9(9) Å^3	
Z	2	
F(000)	426	
Denisty (calculated)	1.53 g cm^{-3}	
Denisty (measured)[2]	1.59(5) g cm^{-3}	
Linear attentuation coefficient (MoK_α)	15.35 cm^{-1}	

Data Collection

Diffractometer	Rigaku AFC6S
Radiation	MoK_α, graphite monochromated
	λ = 0.7107 Å
Scan type	ω-2θ
Scan rate	2.0° min^{-1} in ω
Scan width	(0.95 + 0.30 tan θ)°
2θ range for data collection	5.38 to 50.00°
Reflections collected	3338
Independent reflections	3141
Transmission coefficients[3]	0.80(min), 1.000(max), 0.94(average)
Data corrections	Lorentz-polarisation, absorption correction[4]
Intensity decay of standard reflx	0.5% (3 reflxns checked every 150 data)
Index ranges	$-11 \le h \le 10$, $-13 \le k \le 12$, $0 \le l \le 11$

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[1] Unit cell parameters and their esd's were determined from a least squares fitting of the setting angles of 20 reflections in the range 14.60°≤2θ≤18.84°.

[2] Determined by flotation in $CHBr_3$/hexane mixture (average of three determinations).

[3] Determined from azimuthal scans.

[4] Empirical correction based on azimuthal scans of two reflections.

Structure Solution

Patterson methods with SAPI91[5] and expanded using Fourier techniques with DIRDIF[6]

Refinement Method

Full-matrix least-squares on F^2 with SHELXL93[7] All non-hydrogen atoms were refined anisotropically.

Hydrogen atoms were refined using a rigid model C_{sp2}-H = 0.93Å, $C_{sp3}(CH_3)$-H = 0.96Å, $C_{sp3}(CRH_2)$-H = 0.97 Å, $C_{sp3}(CHR_2)$-H = 0.98 Å, N_{sp3}-H=0.90 Å, O_{sp3}-H = 0.82 Å with $U_{iso}[H(C_{sp2})] = 1.2U_{eq}(C_{sp2})$, $U_{iso}\{H[C_{sp3}(CH_3)\text{-}H]\} = 1.5U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CRH_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}\{H[C_{sp3}(CHR_2)\text{-}H]\} = 1.2U_{eq}(C_{sp3})$, $U_{iso}[H(N_{sp3}\text{-}H)] = 1.2U_{eq}(N_{sp3})$ and $U_{iso}[H(O_{sp3}\text{-}H)] = 1.5U_{eq}(O_{sp3})$

Weighting scheme	$w = [\sigma^2(F_o^2) + (0.0366P)^2 + 0.24P]^{-1}$
	$P = [\max(I_{obs}, 0) + 2F_c^2] / 3$
Data[8] / restraints / parameters	3140 / 0 / 210
Data to parameter ratio	14.95
Goodness-of-fit[9] on F^2	1.05
R(%)[10] indices	
$[I_o > 2\sigma(I_o)]$ 2409 data	$R1 = 3.20$, $wR2 = 7.33$
All 3338 data	$R1 = 5.70$, $wR2 = 8.57$
Final difference map	
largest diff. peak[11] and hole	0.27 and -0.22 eÅ^{-3}
rms deviation from mean	0.05 eÅ^{-3}
Largest shift/ esd in final cycle	0.00

[5] F. Hai-Fu. Structure analysis programs with intelligent control, Rigaku Corporation, Tokyo, Japan, 1993.
[6] P. T. Beurskens, G. Admiraal, G. Beurskens, G. Bosman, W. P. Garcia-Granda, R. O. Gould, J. M. M. Smits and C. Smykalla. The DIRDIF program system, Technical report of the crystallography laboratory, University of Nijmegen, The Netherlands, 1992.
[7] G. M. Sheldrick, SHELXL93. Program for crystal structure refinement. Univ. of Gottingen, Germany.
[8] One reflection suppressed in the refinement.
[9] GooF = $\{\Sigma[w(F_o^2 - F_c^2)^2] / (n\text{-}p)\}^{1/2}$ p = number of parameters, n = number of data.
[10] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR2 = \{\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$
[11] Located 1.05□ away from Cl2.