

Figure out what is A2.

A Schiff Base ligand was synthesized by reacting salicylaldehyde (C<sub>7</sub>H<sub>6</sub>O<sub>2</sub>) and one of two different diamines (C<sub>2</sub>N<sub>2</sub>H<sub>8</sub> or C<sub>3</sub>N<sub>2</sub>H<sub>10</sub>). The resulting crystalline product underwent IR analysis, UV-Vis analysis and melting point tests, and data collated. A portion of the ligand was further reacted with Cu<sup>2+</sup> to create a Cu complex of the ligand, which also underwent UV-Vis and IR analysis. The combined data was compared to accredited NMR/Mass spectrum data and the chemical and structural formula of the Cu complex, the Schiff Base Ligand and original diamine were derived.

Experimental:

Synth of Ligand:

Clear and colourless Salicylaldehyde (10ml, 11.46g,  $9.38 \times 10^{-2}$  mol) was mixed with the clear and colourless diamine (A2, 10ml) which immediately reacted into a clear bright yellow solution. This solution was heated for 15min to ensure a complete reaction, then cooled on a ice bath. With slight scratching clouds of crystals soon precipitated, and were removed from solution with a Buchner funnel. The crystals were washed with ice-cold EtOH (95%, 5ml), thoroughly dried and weighed (Yield xxg).

Synth of Cu<sup>2+</sup> Complex:

The prepared Schiff Base Ligand (0.5g) was dissolved in EtOH (95%, 20mL) and Cupric acetate (37.5g, 0.2 mol) added. Solution was heated lightly for 20min, and allowed to cool slowly to room temperature. The solution was left for 7 days, and the resulting dark blue/black needle-like crystals filtered of in Buchner funnel. Cupric acetate crystals were noted in product, so re-dissolved in EtOH (95%, 20ml) and crashed crystals out again with an ice bath. Deep-blue Cupric acetate crystals (~.05% mass) were still noted, but minimal compared to total yield of complex (0.38g)

Analysis of Ligand:

IR spectrum: A small amount of the ligand (approx 1mm<sup>3</sup>) was mullied up with Nujol and spectrum recorded, noting the peaks consistent for a Nujol mix.

UV-Vis: A small amount of the ligand (approx 1mm<sup>3</sup>) was dissolved in acetone (10ml) and Vis spectrum recorded, machine calibrated to remove acetone noise from results.

Melting point: The melting point of the ligand was recorded at 325.62 K on one machine, and 335.62 K on the other. Results were consistent depending on which machine was used.

Analysis of Cupric Complex:

UV-Vis and IR spectrum were recorded as per standard lab practice (detailed as above for ligand). Melting point was not analyzed, as would have likely been out of machine's range.

Results:

MP of Ligand: 325.62 +/- 4 K, or 335.62 +/- 4K depending if using machine 1 or 2.

## Mass Spectrum

Comparing the highest m/z of A1 (269.1281) and A2 (283.1449) shows a difference of 14.0168, or one CH<sub>2</sub> group (or possibly a single N). Subtracting the weight of 2x N substituted Salicaldehyde leaves ~29 and ~43 respectively, or possibly 2x CH<sub>2</sub>, 3x CH<sub>2</sub> (+ mystery proton!)

A1 Ligand		A2 Ligand	
m/z 269.1281	M+, or total molecular weight	m/z 283.1449	M+, or total molecular weight
m/z 223.0861 (very low %)	M+-46.042	m/z 254.117	M+-29.0279
m/z 187.0850	M+-82.0431	m/z 240.1026	M+-43.0423
m/z 165.1032	M+-104.0249	m/z 201.0996	M+-82.0453
m/z 148.0764	M+-121.0517	m/z 189.10	M+-94.0449
m/z 131.0497	M+-138.0784	m/z 179.1189	M+-104.026
m/z 122.0607	M+-147.0674	m/z 162.0924	M+-121.0525
m/z 107.0497	M+-162.0784	m/z 148.0766	M+-135.0683
		m/z 136.0766	M+-147.0683
		m/z 122.0608	M+-161.0841
		m/z 107.0493	M+-176.0956

A1 Cu Complex		A2 Cu Complex	
m/z 330.0474	M+, or total molecular weight	m/z 344.0612	M+, or total molecular weight
m/z 312.1026	M+-17.9448	m/z 114.9825	M+-229.0787
m/z 289.1196	M+-40.9278		
m/z 242.2893	M+-87.7581		
m/z 130.9811	M+-199.0663		
m/z 113.9678	M+-216.0796		

## Empirical Data:

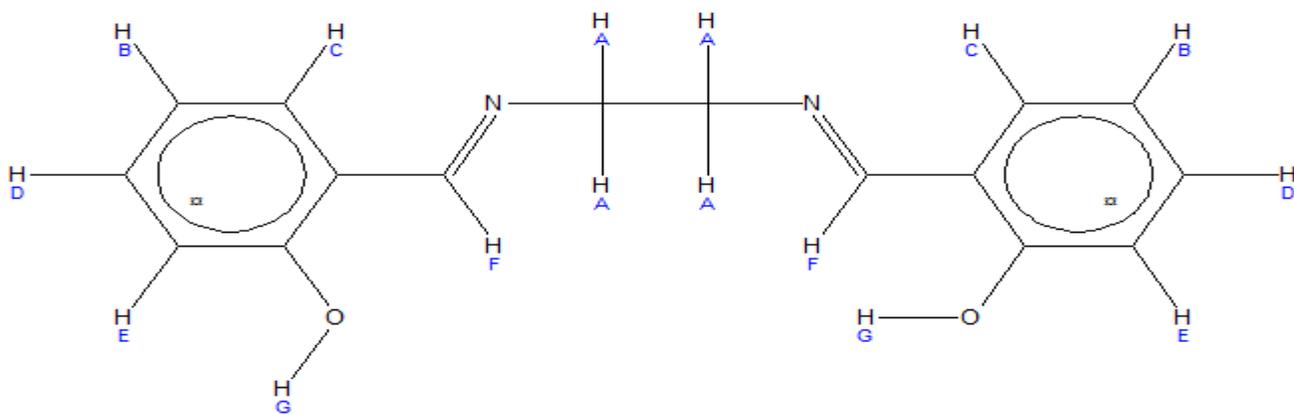
Following suggested formulas calculated by  $\sim ([A]\% \times (M+) / [A]u)$   
 where A= C, H or N, M+ = total molecular weight, u = atomic weight

A1 Ligand:	Suggested formula	A2 Ligand:	Suggested formula
C 71.81%, H 6.11%, N 10.52%+(11.56%)	C16,H16,N2+(O2)	C 72.17%, H 6.51%, N 9.98%+(11.34%)	C17,H18,N2+(O2)
A1 Cu Complex:		A2 Cu Complex:	
C 58.62%, H 4.46%, N 8.43%+(28.49%)	C16,H14,N2+(O2,Cu)	C 59.23%, H 4.81%, N 8.06%+(27.9%)	C17,H16,N2+(O2,Cu)

NMR:

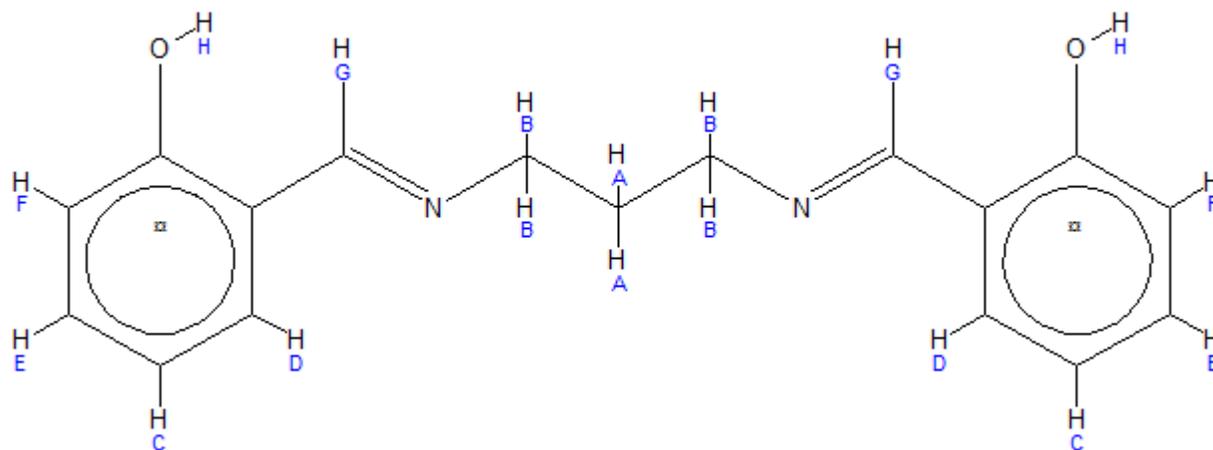
A1

H assign	/ppm	Peak Area	No. of Lines	Nearest Neighbors	Neighbor Assignment
A	4	4	1	0	N=[C]-C=N
B	6.85	2	3	2	N=C-C-C-[C]
C	6.95	2	2	1	N=C-C-[C]
D	7.25	2	2	1	HO-C-C-[C]
E	7.35	2	3	2	HO-C-[C]
F	8.4	2	1	0	N=[C]-C
G	13.2	2	1	0	[O]-C



A2

H assign	/ppm				
A	2.15	2	5	4	N=C-[C]-C=N
B	3.75	4	3	2	N=[C]-C-[C]=N
C	6.9	2	3	2	N=C-C-C-[C]
D	7	2	2	1	N=C-C-[C]
E	7.25	2	2	1	HO-C-C-[C]
F	7.35	2	3	2	HO-C-[C]
G	8.4	2	1	0	N=[C]-C
H	13.5	2	1	0	[O]-C



IR Absorption (discounting peaks associated with nugol mix):

A2 Ligand Peaks:	Attribute:	A2 Cu Complex:	Attribute:
1614	C-H (ring)	1537	?
1632	C-OH Stretch	1614	C-H (ring)
1279.9	C-O stretch	2358	?
2800-2900	C-H stretch	2800-2900	C-H stretch

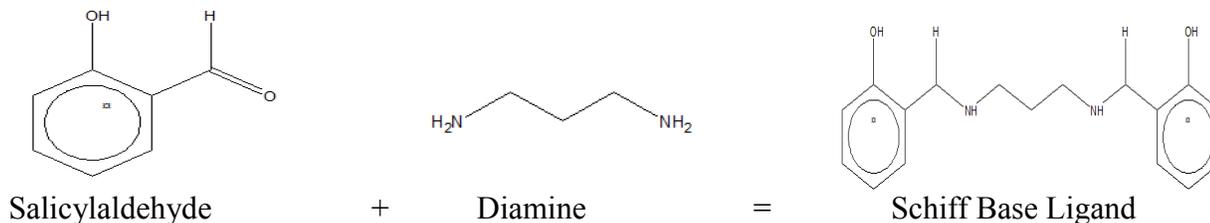
UV-Vis

A2 Ligand Peaks:		A2 Cu Complex:	
610nm	0.3AU	610nm	0.4AU
460nm	0.3AU	460nm	0.3AU
450nm	0.4AU	450nm	0.5AU
440nm	1.0AU	440nm	0.7AU
430nm	1.7AU	430nm	0.9AU
420nm	2.0AU	420nm	1.5AU
410nm	2.4AU	410nm	4.0AU
400nm	2.4AU	400nm	2.7AU

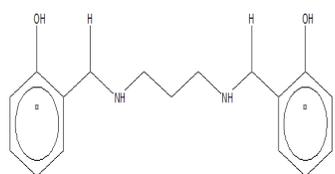
Noted are the difference at 610nm, small due to dilution of solution (Red-Orange absorbency), Slow peaks from 450m-360nm, more pronounced in the Cu complex with spike at 408nm.

Discussion:

Synth of ligand:

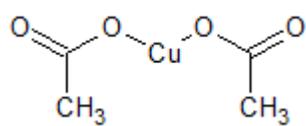


Synth of Complex:

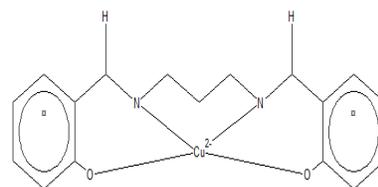


Schiff Base Ligand

+



Cupric acetate



= Cu Complex