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# Norbornenyl Aroylhydrazones and Their Copper (I) and Mercury (I) Complexes

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# NORBORNENYL AROYLHYDRAZONES AND THEIR COPPER(I) AND MERCURY(I) COMPLEXES

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### ABSTRACT

Seven new aroyl hydrazones containing the norbornene group and their copper(I) and mercury(I) complexes have been synthesized and characterized by elemental analyses, electrolytic conductance, IR spectra, <sup>1</sup>H NMR spectra and TGA analyses. These ligands coordinate to metal ions in the enolic form and act as bidentate ligands. All complexes are non-electrolytes in methanol-N,N-dimethylformamide (3 : 1 in volume).

#### INTRODUCTION

Characteristic properties and bioactivities of norbornene derivatives have been investigated extensively, including their use in the synthesis of heterocyclic compounds<sup>1-6</sup> and as inhibitors for microorganisms, e. g. antimicrobial, bactericidal<sup>7</sup>, fungicidal<sup>8</sup>; as herbicides and antidotes<sup>9</sup>. But the arylhydrazones containing norbornenyl as well as their transition metal complexes have not been reported so far.

In the present communication we report the synthesis and characterization of the 2-acetyl-3, 3-dimethyl bicyclo [2, 2, 1] hept-5-ene aroylhydrazones (HL<sup>1-7</sup>) in Fig. 1 and their copper(I) and mercury(I) complexes.

#### **EXPERIMENTAL**

2-Acetyl-3, 3-dimethylbicyclo [2, 2, 1] hept-5-ene was prepared by the method given in the literature<sup>4</sup>. Other reagents used were of analytical reagent grade.

#### **Physical Measurements**

All physical measurements were carried out by routine literature procedures. <sup>10</sup>

### Syntheses of Hydrazones

All hydrazones  $(HL^{1-7})$  were prepared using the same procedure, e. g. a mixture of 1.64 g (10 mmol) 2-acetyl-3,3-dimethyl bicyclo[2,2,1]hept-5-ene in 5 mL 1-propanol and 1.90 g (10.5 mmol) *p*-nitrobenzoyl hydrazide in 30 mL 1-propanol was refluxed for 12 h with stirring. The solution was concentrated to 15 mL and cooled to 0-5°C, the precipitated crystals were collected on a filter, washed two times with ether, alcohol and water, respectively, recrystallized from ethanol—water (3 : 1 in volume), and dried *in vacuo* at room temperature. The ligand HL<sup>6</sup> was obtained in 2.66 g yield, 81.3%.



Figure 1. Structures of the Ligands.

#### Preparation of Copper(I) and Mercury(I) Complexes

All complexes 8 - 16 were prepared by the same method. A solution of copper(I) acetate dihydrate (0.10 g, 0.5 mmol) in alcohol (8 mL) was added dropwise to (0.29 g, 1 mmol) 2-acetyl-3, 3-dimethyl bicyclo[2, 2, 1]hept-5ene benzoyl hydrazone (HL<sup>1</sup>) in alcohol (10 mL) and refluxed for 2 h with stirring. After cooling to room temperature, the resulting precipitate was collected on a filter, washed twice with ethanol and ether, and dried. Yield: 0.24 g (76.7%). The elemental analyses and physical properties of the ligands and their complexes are listed in Table I.

All complexes are slightly soluble in methanol, ethanol, acetone, ether and chloroform and readly soluble in DMF and DMSO. The solutions of the copper-

No.	Formula	M. P. (°C)	Color	Yield	$\Lambda_{m}$	Found(Calcd.)%		
	(Formula Weight)	(Dec. P. )		%	$\Omega^{-1} cm^2 mol^{-1}$	С	н	N
1	HL <sup>1</sup> C <sub>18</sub> H <sub>22</sub> N <sub>2</sub> O(282. 4)	136-138	white	69.2		76.46 (76.56)	7.85 (7.91)	9.94 (9.92)
2	HL <sup>2</sup> C <sub>16</sub> H <sub>25</sub> N <sub>2</sub> O <sub>2</sub> (273. 3)	178-179	white	74.4	-	70.68 (70.56)	7.44 (7.40)	10.30 (10.29)
3	HL <sup>3</sup> C <sub>18</sub> H <sub>20</sub> Cl <sub>2</sub> N <sub>2</sub> O(351.3)	173-175	white	77.8	-	61.53 (61.54)	5.74 (5.74)	8.11 (7.98)
4	HL <sup>4</sup> C <sub>17</sub> H <sub>21</sub> N <sub>3</sub> O(283.4)	114-116	white	59.1		72.14 (72.05)	7.56 (7.47)	14.68 (14.83)
5	HL <sup>5</sup> C <sub>18</sub> H <sub>21</sub> N <sub>3</sub> O <sub>3</sub> (327. 4)	151-153	pale brown	84.5	-	65.94 (66.03)	6.45 (6.47)	12.78 (12.84)
6	HL <sup>6</sup> C <sub>18</sub> H <sub>21</sub> N <sub>3</sub> O <sub>3</sub> (327. 4)	151-153	pale brown	81.3		65.82 (66.03)	6.49 (6.47)	12.79 (12.84)
7	HL <sup>7</sup> C <sub>19</sub> H <sub>24</sub> N <sub>2</sub> O <sub>2</sub> (312. 4)	154-156	white	76.6		72.82 (73.04)	7.74 (7.74)	8.96 (8.97)
8	$Cu(L^1)_2$ $C_{36}H_{42}CuN_4O_2(626.3)$	(228)	pale green	76.7	12.0	68.51 (69.04)	6.74 (6.76)	9.18 (8.94)
9	$Cu(L^2)_2 \cdot H_2O$ $C_{32}H_{40}CuN_4O_5(624.2)$	(180)	green	73.7	13.1	62.10 (61.57)	6.33 (6.46)	8.96 (8.97)
10	$Cu(L^3)_2$ $C_{36}H_{38}Cl_4CuN_4O_2(764.0)$	) (198)	brown	79.0	12.0	56.38 (56.59)	6.43 (6.42)	7.30 (7.33)
11	$Cu(L^4)_2 \cdot H_2O$ $C_{34}H_{40}CuN_5O_2(628.3)$	(199)	green	81.0	28.3	62.94 (63.18)	6.39 (6.55)	12.85 (13.01)
12	$Cu(L^5)_2$ $C_{36}H_{40}CuN_6O_6(716.3)$	(210)	brown	83.9	39.2	60.28 (60.36)	5.60 (5.63)	11.42 (11.74)
13	Cu(L <sup>6</sup> ) <sub>2</sub> C <sub>36</sub> H <sub>40</sub> CuN <sub>6</sub> O <sub>6</sub> (716.3)	(248)	pale brown	82.3	23. 5	59.94 (60.36)	5.61 (5.63)	11.45 (11.74)
14	$Cu(L^7)_2$ $C_{38}H_{46}CuN_4O_4(686.3)$	(234)	pale green	84.4	14.9	66.32 (66.49)	6.70 (6.76)	8.38 (8.16)
15	$Hg(L^3)_2 \cdot H_2O$ $C_{36}H_{40}Cl_4HgN_4O_3(919.1)$	) (235)	pale red	72.6	25.7	47.28 (47.04)	4.52 (4.39)	5.96 (6.10)
16	$Hg(L^{7})_{2}$ C <sub>38</sub> H <sub>46</sub> HgN <sub>4</sub> O <sub>4</sub> (823. 4)	(233)	yellow	73.1	21.9	55.21 (55.43)	5.49 (5.63)	6.76 (6.81)

Table 1. Analytical Data of the Ligands and Their Complexes.

(I) complexes in methanol appeared pale green to deep green, the solutions of the mercury (I) complexes in DMF are pale red to yellow. All compounds, whether ligands or chelates, are stable in air.

### **RESULTS AND DISCUSSION**

The ligands 1-7 have been obtained by the condensation reaction of 2-acetyl-3, 3-dimethylbicyclo[2, 2, 1]hept-5-ene with aroylhydrazide as shown in equation (1).

$$+ H_2 NNHCOAr \rightarrow (1)$$

These ligands can exist in two forms, *i. e.* the keto (A) or enolic form (B).

$$\begin{array}{cccc} \mathbf{C}\mathbf{H}_{3} & \mathbf{O} & \mathbf{C}\mathbf{H}_{3} & \mathbf{O}-\mathbf{H} \\ & & & & \\ \mathbf{C}_{9}\mathbf{H}_{13}-\mathbf{C}=\mathbf{N}-\mathbf{N}\mathbf{H}-\mathbf{C}-\mathbf{A}\mathbf{r} & (\mathbf{A}) \Longrightarrow & \mathbf{C}_{9}\mathbf{H}_{13}-\mathbf{C}=\mathbf{N}-\mathbf{N}=\mathbf{C}-\mathbf{A}\mathbf{r} & (\mathbf{B}) \end{array}$$
(2)

When using metal acetates, the ligand coordinates to metal ions in the enolic form (B) as shown in equation (3).

$$2 \xrightarrow{\mathbf{R}} \mathbf{N} - \mathbf{N} \xrightarrow{\mathbf{O} - \mathbf{H}}_{\mathbf{Ar}} + \mathbf{M}(\mathbf{AcO})_2 \rightarrow \mathbf{M} \left[ \begin{array}{c} \mathbf{R} & \mathbf{O} \\ \mathbf{R}' & \mathbf{N} - \mathbf{N} = \mathbf{O} \\ \mathbf{R}' & \mathbf{Ar} \end{array} \right]_2 + 2\mathbf{HOAc} \quad (3)$$
$$\mathbf{R} = \mathbf{CH}_3; \qquad \mathbf{R}' = \mathbf{C}_9 \mathbf{H}_{13}$$

#### IR Spectra

The IR frequencies of the ligands and their complexes along with their assignments are given in Table I. The hydrazone bands appearing around 3183, 1656, 1608 and 923 cm<sup>-1</sup> are attributed to  $\nu(N-H)$ ,  $\nu(C=O)$ ,  $\nu(C=N)$  and  $\nu(N-N)$ , respectively. The presence of these bands suggest that these compounds exist predominantly in the keto form (A) in the solid state. The IR spectra of the copper(I) and mercury(I) complexes show significant changes

No	v(N-H)	v(C=O)	v(C=N-N=C)	v(C=N)	(NCO <sup>-</sup> )	v(N-N)	v(M-O)	v(M-N)
1	3194 br, s	1644 vs		1626 vs	-	932 m	-	_
2	3185 s	1664 vs	—	1610 sh	_	926 m	_	
3	3174 m	1667 vs		1591 m		925 w	-	—
4	3192 br. s	1664 vs	_	1602 s		922 w	-	
5	3181 m	1650 vs	-	1615 m	_	908 w	-	_
6	3172 m	1662 vs	-	1601 s		914 w	-	
7	3354 m	1657 vs	-	1606 vs		938 w	-	-
8			1610 sh	1593 s	1374 vs	946 w	410 w	282 w
9	-	-	1606 vs	1576 w	1352 s	953 w	429 w	300 w
10		-	1584 vs	1566 vs	1364 s	945 m	415 w	293 w
11	-	-	1626 vs	1535 vs	1370 vs	950 w	436 w	266 w
12	_	_	1615 w	1592 s	1368 s	958 w	409 w	292 w
13		-	1600 sh	1581 s	1369 s	948 w	410 w	279 w
14		-	1608 vs	1591 vs	1375 vs	945 w	405 w	255 w
15	-	-	1617 sh	1578 s	1361 s	963 <b>w</b>	421 w	290 w
16	-	-	1601 vs	1585 s	1379 m	944 w	421 w	263 w

Table I . Important IR Absorption Bands (cm<sup>-1</sup>) of the Ligands and Their Complexes.

15 - - 1617 sh 1578 s 1361 s 963 w 421 w 290 w 16 - - 1601 vs 1585 s 1379 m 944 w 421 w 263 w as compared with those of the parent ligands. The bands due to  $\nu(N-H)$  and  $\nu$ (C=O) are absent in these complexes. Two new very strong bands observed around 1608 and 1368 cm<sup>-1</sup> can be assigned to  $\nu$  (C = N - N = C) and  $\nu$ (NCO<sup>-</sup>)<sup>11, 12</sup> which are due to the enolization of the carbonyl group<sup>13</sup>. The  $\nu$ (C = N) band is found to shift to lower frequency by *ca*. 15-25 cm<sup>-1</sup> whereas the  $\nu$ (N-N) band moves to higher frequency by *ca*. 7-44 cm<sup>-1</sup> indicating that the azomethine nitrogen is involved in bonding<sup>14</sup>. Two new bands at 405-436 cm<sup>-1</sup> and 255-300 cm<sup>-1</sup> are attributed to  $\nu$ (M-O) and  $\nu$ (M-N)<sup>15</sup>, respectively,

which are not observed in the spectra of the free ligands. These changes clearly

indicate that the ligands enolize in solution and chelate to the metal ion through the azomethine nitrogen atom and the enolic oxygen atom. The hydrazones act as uninegtive bidentate ligands.

#### H NMR Spectra

The <sup>1</sup>H NMR spectra data of the ligands (1-7) and the mercury (I) complexes (15 and 16) are given in Table II. The ligand spectra are similar in the 0.80-6.30 ppm region. The chemical shift of the protons on the *gem*-dimethyl of position 3 of the norbornenyl moiety are exhibited at  $\delta = 0.93 - 1.05$  ppm, 1.00-1.11 ppm and  $\delta = 0.70-0.80$  ppm, 1.31-1.40 ppm and are attributable to exo-form and endo-form protons<sup>3</sup>, respectively. It is shown that these ligands exist predominantly in the exo-form (exo : endo = 2-6 : 1). A broad signal due to the N-H proton is observed at 8.46-9.54 ppm, which disappears on deuteration.

It can be seen that the proton signals of N-H disappear in the mercury-(I) complexes, while other proton signals do not show significant changes as compared with that of the ligands. It is suggested that the carbonyl group coordinates to the central ion through keto-enol tautomerization and deprotonation. This is consistent with the results of the IR spectra of these compounds. Normally, clear <sup>1</sup>H NMR spectra of the copper(I) complexes can not be obtained because of the interference of the copper(I) ion due to its paramagnetic properties.

#### Thermal Analyses

There are two remarkable features in the TGA data (Table N). First, the melting points (or decomposition temperatures) of the complexes are higher than those of the ligands indicating that the complexes are more stable than the

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<sup>1</sup>H NMR Chemical Shifts ( $\delta$ , ppm) of Ligands and Mercury Complexes. Table II.

	~		and the second			
6. 18 1. 97 0. 79–1. 33		2.95	2. 15	2.31	0.80-1.80	7.31-7.96 <sup>b</sup>
n, 2H) (s, 3H) (m, 6H)		(d,1H)	(m, 1H)	(m,1H)	(m,2H)	(m,5H)
6.20 1.96 0.801.39		3.07	2, 15	2.36	0.83-1.80	6.49-7.49°
n,2H) (s,3H) (m,6H)		(d,1H)	(m, 1H)	(m,1H)	(m,2H)	(m,3H)
6.18 1.91 0.70-1.31		2.52	2.02	2.22	0.82-1.76	7.29-7.45
n,2H) (s,3H) (m,6H)		(d.1H)	(m,1H)	(m,1H)	(m,2H)	(m,3H)
6.17 2.00 0.77-1.38		2.77	2.30	2.44	0.80-2.10	7.67-8.67
n,2H) (s,3H) (m,6H)		(d,1H)	(m,1H)	(m,1H)	(m,2H)	(m,3H)
6.17 2.06 0.80-1.40		2.85	1.92	2.30	0.83-1.85	7.40-8.80
n.2H) (s.3H) (m.6H)		(d.1H)	(m,1H)	(m,1H)	(m.2H)	(m,3H)
6.19 2.04 0.80-1.40		2.77	2.15	2.38	0.85-1.85	7.95-8.30
n,2H) (s,3H) (m,6H)		(d,1H)	(m,1H)	(m,1H)	(m,2H)	(m,3H)
6.18     1.96     0.791.38       n,2H)     (s,3H)     (m,6H)		2.96 (d.1H)	2.10 (m,1H)	2.31 (m,111)	0.82-1.76 (m,2H)	6.80-7.44 (m,4H)
6.15 1.84 0.70-1.20		2.92	2.10	2. 32	0.68-1.68	7.28-7.68
n,4H) (s,6H) (m,12H)		(d,2H)	(m,2H)	(m,2H)	(m.4H)	(m,6H)
6.20 1.90 0.88-1.20 n,4H) (s,6H) (m,12H)		2.90 (4.94)	2.00 (m.2H)	2.30 ( 31)	0.78-1.76	6.86-7.92 (m.8H)

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a. Disappears on deuterium exchange; b.  $CDCl_3$  as a solvent for 1-7 and  $DMSO-d_6$  for 15 and 16; c. Ar is furcyl.

Compound	D.T.(C)	Leaving group	Weight loss (%)*
2	182-230	C <sub>s</sub> H <sub>6</sub>	23.3 (24.2)
	230-252	$C_4H_3O-CONHN =$	47.4 (45.5)
	252-540	$(CH_3)_2C = CH - C(CH_3) =$	26.3 (30.1)
	>540	residue	3.0 (0.0)
9	135-175	H <sub>2</sub> O	3.0 (2.9)
	212-250	2 N <sub>2</sub>	8.8 (9.0)
	250-285	2 C <sub>5</sub> H <sub>6</sub>	21.0 (21.2)
	<b>285 - 4</b> 50	$2 (CH_3)_2 C = CH - C(CH_3) =$	24.7 (26.3)
	450-555	2 C4H3O-CO	29.8 (30.5)
	>555	CuO	13.0 (12.8)
6	180-260	$C_{5}H_{6}$ , $-NH-N =$	26.2 (29.1)
	260-445	$(CH_3)_2C = CH - C(CH_3) =$	23.0 (25.1)
	445-625	O <sub>2</sub> N-C <sub>5</sub> H <sub>4</sub> -CO	47.8 (45.8)
	>625	residue	3.0 (0.0)
13	247-344	2 C <sub>5</sub> H <sub>6</sub>	19.5 (18.5)
	344-375	2 N <sub>2</sub>	7.5 (7.8)
	375-503	$2 (CH_3)_2 C = CH - C(CH_3) =$	21.8 (22.9)
	503-570	$2 O_2 N - C_4 H_4 - CO$	40.6 (41.9)
	>570	CuO	10.6(11.1)

Table  $\mathbb{N}$ . The Data of TGA Analyses.

\* Calculated values are given in parentheses.



Figure 2. Proposed Structure of the Complexes; M = Cu(I), Hg(I).

ligands. The second is that all compounds give off cyclopentadiene by the retro Diels-Alder reaction. Then they release nitrogen and another part of the bridging ring  $[(CH_3)_2C=CH-C(CH_3)=]$ . Compound 9 decompose at 135-175°C to release a water molecule which perhaps is a coordinated one.

#### Molar Conductance

The molar conductances of all complexes 8-16 (Table I) were measured in the region as 16. 8-36. 5 ohm<sup>-1</sup> cm<sup>2</sup> mol<sup>-1</sup> (at 25°C), indicating their nonelectrolytic nature<sup>16</sup>.

# CONCLUSIONS

All of above mentioned results indicate that the ligands are enolized in solution and coordinate to metal ions through the azomethine nitrogen atom and enolic oxygen atom. The hydrazone acts as an uninegative bidentate ligand as shown in Fig. 2.

### ACKNOWLEDGEMENT

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