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# Reactions of $MoCl_5$ with Succinimide, Imidazole, 3-Methylpyridine and 4-Methylpyridine in Tetrahydrofuran

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**ABSTRACT**

$MoCl_5$ was reacted with succinimide/imidazole/3-methylpyridine/4-methylpyridine in THF medium using equal/double molar concentrations of the ligand at room temperature. The end products obtained are: $MoO_2Cl_4(C_4H_5NO_2)_4(C_4H_8O)$,[1]; $Mo_2O_{12}Cl_7(C_3H_4N_2)_7$, [2]; $Mo_2O_{12}Cl_7(C_3H_4N_2)_7$, [3] and $Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2$, [4]. The above compounds were characterized by FTIR, $^1$H NMR, $^{13}$C NMR, LC-MS, microbiological and C, H, N, Mo, Cl studies. All procedures and work outs were handled in vacuum line using dry nitrogen atmosphere to protect the products from oxidation/ hydrolysis by air/moisture. Elemental data and fragments visualized in LC-MS are concordant with the formulae derived.

**Keywords:** Succinimide, Imidazole, 3-methylpyridine, 4-methylpyridine, $MoCl_5$, FTIR, $^1$H NMR, $^{13}$C NMR, DMSO-$d_6$, LC-MS, microbiological, THF medium.

## INTRODUCTION

### Succinimide

Succinimides[1] are used as precursors for biological applications. Succinimide is a part of various biologically active molecules having properties: antitumour[2], CNS depressant[3], anorectic[4], hypotensive[5], analgesic[6], cytostatic[7], nerve conduction blocking[8], antispasmodic[9], bacteriostatic[10], muscle relaxant[11], antibacterial[12], antifungal[13], anti-convulsant[14] and anti-tubercular[15].

### Imidazole

Imidazole containing drugs are used[16-28] as: anticoagulants, 20-carboxypeptidase inhibitors, antifungal, β-lactamase inhibitors, hemeoxygenase inhibitors, anticancer, antitubercular, anti-inflammatory, antibacterial, antiviral, antidiabetic HETE (20-Hydroxy-5,8,11,14-eicosatetraenoic acid) synthase inhibitors, antimalarial and antiaging agents.

### 3-Methylpyridine

3-Methylpyridine[29] is used to prepare agrochemical chlorpyrifos[30]. As compared to 2-methylpyridine/4-methylpyridine[31,32] there is low degradation and poor volatility of 3-methylpyridine from water samples. 3-Methylpyridine is used as

an antidote for organophosphate poisoning[33]. It is biodegradable.

**4-Methylpyridine**

Many heterocyclic compounds can be prepared from 4-methylpyridine[34]. It is a precursor to other commercially significant species, often of medicinal interest. 4-Methylpyridine is a precursor for the preparation of the antituberculosis drug[35] 'isoniazid'. It is very reliable and commonly used medicine for tuberculosis.

It has been noted that many drugs have greater activity as metal chelates as compared to organic compounds[36-41].

**AIM of investigation**

Molybdenum(V) chloride has been reported to react with a variety of N-heterocyclic bases. Many reactions of aromatic azoles, diaminoalkanes, imides, 4-phenylimidazole-2-thiol, alkylpyridines, 2-thiazoline-2-thiol, mercaptopyridine-N-oxide sodium and thiols with $MoCl_5$ have been studied[42-47] by the author.

In view of the fact that complexes of these bases with transition metals show various applications complexes of succinimide, imidazole, 3-methylpyridine and 4-methylpyridine with $MoCl_5$ have been synthesized and studied. Characterization of these complexes was executed with $^1H$ NMR, $^{13}C$ NMR, FTIR, LC-MS, microbiological studies and elemental analysis.

## MATERIALS AND METHODS

Succinimide, imidazole, 3-methylpyridine, 4-methylpyridine and $MoCl_5$ were purchased from Sigma-Aldrich.

The products are easily oxidized/hydrolysed by air/moisture, so all procedures and work outs were handled in vacuum line using dry nitrogen atmosphere to protect the products from oxidation/ hydrolysis by air/moisture.

Ligand dissolved in dry THF was combined from dropping funnel with $MoCl_5$ dropwise with continuous agitation. The reaction was carried out for 7-8 hours. Filtration unit fitted with G-4 sintered glass crucible was used for filtration and isolation of products.

Molybdenum analysis was performed by oxinate method gravimetrically[48]. Chlorine analysis was performed by silver chloride method gravimetrically[48]. Thermo Finnigan Elemental Analyser was used for analysis of remaining elements. Perkin-Elmer 400 FTIR Spectrometer was used for obtaining vibrational spectra. $^1H/^{13}C$ nuclear magnetic resonance spectra in DMSO-$d_6$ were obtained with Multinuclear Brucker Avance-II 400 NMR spectrometer. LC-MS spectra were obtained in the range 0-1100 m/z. Above instruments were used at P. U. Chandigarh.

Antibacterial and antifungal activities of molybdenum compounds synthesized were tested using strains: *Gram-positive* bacteria *Staphylococcus aureus* (MTCC-737), *Gram-negative* bacteria *E. coli* (MTCC-1687), Fungi *Candida albicans* (MTCC-227) and *Aspergillus niger* (MTCC-282). Agar well diffusion assay method was used. Standard drug (amoxicillin) for bacteria and standard drug (ketoconazole) for virus as reference were used. MTCC (The Microbial Type Culture Collection and Gene Bank, Chandigarh, India) cultures were used. Testing was carried out at ISF Analytical Laboratory (ISF College of Pharmacy), Ferozepur Road, Moga, Punjab, India.

**Reactions**

(R)/(F) represent the product source,

$$MoCl_5 + C_4H_5NO_2 \xrightarrow[1:2]{THF} MoO_2Cl_4(C_4H_5NO_2)_4(C_4H_8O), [1]$$

Succinimide Black (R)

$$MoCl_5 + C_3H_4N_2 \xrightarrow[1:2]{THF} Mo_2O_{12}Cl_7(C_3H_4N_2)_7, [2]$$

Imidazole Yellowish green (R)

$$MoCl_5 + C_5H_4NCH_3 \xrightarrow[1:2]{THF} Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2, [3]$$

3-Methylpyridine Brown (F)

$$MoCl_5 + C_5H_4NCH_3 \xrightarrow[1:1]{THF} Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2, [4]$$

4-Methylpyridine Dark green (R)

## RESULTS AND DISCUSSIONS

**Elemental Analysis**

Table 1 reveals the percentage of the observed (theoretical) values of the elements.

**Table 1: (Elemental Analysis)**

| Compounds | Cl | Mo | H | C | N |
|---|---|---|---|---|---|
| $MoO_2Cl_4(C_4H_5NO_2)_4(C_4H_8O)$, | 18.77 | 12.10 | 02.57 | 32.37 | 06.85 |
| [1] | (19.24) | (13.00) | (03.25) | (32.52) | (07.58) |
| (Black/738.0) | | | | | |
| $Mo_2O_{12}Cl_7(C_3H_4N_2)_7$, [2] | 21.78 | 16.93 | 02.81 | 22.28 | 17.01 |
| (Yellowish green/1108.5) | (22.41) | (17.32) | (02.52) | (22.73) | (17.68) |
| $Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2$,[3] | 25.57 | 25.53 | 03.88 | 36.07 | 04.68 |
| (Brown/1088.0) | (26.10) | (26.47) | (04.04) | (35.29) | (05.14) |
| $Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2$,[4] | 25.33 | 25.97 | 03.49 | 34.67 | 04.53 |
| (Dark green/1088.0) | (26.10) | (26.47) | (04.04) | (35.29) | (05.14) |

**FTIR Spectra**

Succinimide[49,50] has N-H stretching at 3411 $cm^{-1}$ & 3222 $cm^{-1}$. Band at 3434 $cm^{-1}$ suggests presence of N-H group in [1]. Stretching at 938 $cm^{-1}$ and 918 $cm^{-1}$ reveal the existence of cis-$MoO_2^{2+}$ core[51,52] in [1]. C=O frequency has not altered much from 1773 $cm^{-1}$ and 1711 $cm^{-1}$, indicating thereby absence of Mo–O coordination in [1]. There seems to be little decrease in C=O bond order (Table 2).

**Table 2: (FTIR absorptions in $cm^{-1}$)**

| Assignments | Succinimide[49,50] | [1] |
|---|---|---|
| N-H str. | 3411 s, b, 3222 s, b | 3434.0 s, b |
| $CH_2$ sym. str. | 2962, 2946 w | |
| C=O sym., H-N-C in plane bending | 1773 m | 1773.5 sh |
| C=O asym., H-N-C in plane bending | 1711 vs, b | 1706.1 s |
| $CH_2$ sym. scissoring | 1432 m | 1425.9 sh |
| $CH_2$ asym. scissoring | 1402 m | |
| C-N-C asym. str., H-N-C in plane bending | 1348 s, 1335 | 1371.8 m |
| $CH_2$ bending, ring in plane bending | 1296 s | 1298.1 m |
| $CH_2$ bending | 1242 | 1247.3 sh |
| C-N-C asym. str., H-N-C in plane bending | 1188 s, | 1190.3 s |
| C-C str., CNC sym. str. | 850 | 853.5 sh |
| $CH_2$ bending, ring out of plane bending | 818 s | 818.0 w |
| OCN asym. out of plane bending | 631 m | 643.3 m |
| OCN sym. out of plane bending, $CH_2$ bending | 541 w | 556.3 sh |
| Mo-N str. | | 416.7 w |
| Mo=O Str. of cis-$MoO_2^{2+}$ core[51,52] | | 938.2 w, 918.1 sh |

It is reported that N-H stretching of imidazole[53,54] appears at 3722 $cm^{-1}$ -3272 $cm^{-1}$. There is presence of broad band at 3431 $cm^{-1}$ -3000 $cm^{-1}$ pertaining to N-H group in [2]. This broadening occurs in the solid state (KBr disk) because of hydrogen bonding. A strong band at 975 $cm^{-1}$ is suggestive of terminal Mo=O[51,55,56] in [2] (Table 3).

**Table 3: (FTIR absorptions in $cm^{-1}$)**

| Assignments | Imidazole[53,54] | [2] |
|---|---|---|
| N-H str. | 3722 vb, 3657 vb, 3272, 3242, 3238, 3431.1 vs, 3000 s, vvb | |
| C-H str. | 3195, 3166 | 3157.8 sh, 2997.0 sh |
| C=C ring str. | 1560, 1502 | 1633.2 s, 1592.5 sh |
| N-C ring str. | 1435 | 1445.4 w |
| C-H in plane bending | 1094, 1073 | 1194.1 w, 1079.9 w |
| C-H out of plane bending (wagging), Ring twisting | 817, 728 | 760.7 m |
| Ring twisting | 648 | 627.3 m |
| Ring twisting, N-H wagging | 527 | |
| Terminal Mo=O[51,55,56] str. | | 975.2 m |

C-H ring stretching of 3-methylpyridine[57-60] appears at 3062 $cm^{-1}$ and 3031 $cm^{-1}$. Band at 3055 $cm^{-1}$ has been located in [3]. There is increase of ring C=N str. & ring C=N torsion values and decrease of ring C-H bending mode values, which show presence of some Mo(dπ)→N(pπ) back bonding. A strong band at 977 $cm^{-1}$ is suggestive of terminal Mo=O[45,49,50] in [3] (Table 4).

**Table 4: (FTIR absorptions in $cm^{-1}$)**

| Assignments | 3-Methylpyridine[57-60] | [3] |
|---|---|---|
| C-H Ring str. | 3062, 3031 | 3397.2 s, 3055.8 sh |
| C-H Methyl str. | 3000, 2959, 2926 | 2867.8 sh |
| Ring str. | 1598, 1579 | 1630.5 m, 1552.7 m |
| Ring C-H bending | 1480 | |
| C-H Methyl assym bending | 1457, 1450 | 1469.5 m |
| Ring C-H bending | 1416 | |
| C-H methyl sym. bending | 1386 | 1384.8 w |
| Ring C-H bending | 1363 | 1304.38 sh |
| Ring str. | 1249 | 1263.4 w |
| C-C bond between ring and methyl str. | 1229 | |
| Ring C-H bending | 1192 | 1185.3 sh |
| C-H methyl rocking | 1126, 1045 | 1116.25 m |
| Ring out of plane bending | 1031 | 1044.32 w, 1027.32 w |
| Ring C=N str. | 791 | 890.5 vs, 785.3 m, 737.4 |
| Ring C=N torsion | 712 | 723.6 m |
| Ring bending | 636, 538 | 676.7 m, 511.9 w |
| ∂ C-C bond between ring and methyl | 456 | 464.2 w |
| Terminal Mo=O[45,49,50] str. | | 977.0 w |

C-H ring stretching of 4-methylpyridine[53-60] appears at 3074 $cm^{-1}$ and 3032 $cm^{-1}$. Band at 3097 $cm^{-1}$ has been located in [4]. There is increase of ring C=N str. & ring C=N torsion values and decrease of ring C-H bending mode values, which show presence of some Mo(dπ)→N(pπ) back bonding. A strong band at 976 $cm^{-1}$ is suggestive of terminal Mo=O[45,49,50] in [4] (Table 5).

**Table 5: (FTIR absorptions in $cm^{-1}$)**

| Assignment | 4-Methylpyridine[57-60] | [4] |
|---|---|---|
| C-H Ring str. | 3074, 3032 | 3400.9 vs, b, 3097.5 sh |
| C-H Methyl str. | 2992, 2926 | 2948.8 sh |
| Ring str. | 1610, 1563 | 1640.3 s, 1508.0 m |
| Ring C-H bending | 1501 | |
| C-H Methyl assym bending | 1458, 1445 | 1444.7 sh |
| Ring C-H bending | 1418 | |
| C-H methyl sym. bending | 1383 | 1379.6 w |
| Ring C-H bending | 1365 | 1316.0 w |
| Ring str. | 1227 | 1256.0 w |
| C-C bond between ring and methyl str. | 1210 | 1204.0 w |
| Ring C-H bending | 1194 | |
| C-H Methyl rocking | 1114, 1072 | 1113.4 w, 1039.6 w |
| Ring out of plane bending | 995, 875 | |
| Ring C=N str. | 730 | 793.8 m, 738.0 m |
| Ring C=N torsion | 524 | 569.1 sh |
| ∂ C-C bond between ring and methyl | 486 | 476.5 w |
| Terminal Mo=O[45,49,50] str. | | 976.1 s |

## $^1$H NMR Spectra

$CH_2$ peaks of succinimide[61,62] absorb at 2.74 ppm. $^1$H NMR of [1] in DMSO-$d_6$ shows $CH_2$ peaks at 2.63 ppm showing upfield shift (Table 6). ↑ and ↓ represent upfield/downfield shift.

**Table 6: ($^1$H NMR absorptions in ppm)**

| Assignments | Succinimide[61,62] in $CDCl_3$ | [1] |
|---|---|---|
| N-H | 8.9 | 11.12↓ |
| $CH_2$ | 2.75 | 2.63 ↑ |
| Residual[63] DMSO-$d_6$ | | 2.57 |
| THF[63] C-2, 5 | | 3.51 |
| THF[63] C-3, 4 | | |

Imidazole[64,65] spectrum in CDCl3 shows C-H proton (in middle of nitrogen atoms) absorption at 7.72 ppm. Remaining C-H protons absorb at 7.14 ppm. N-H absorption occurs at 11.60 ppm. Spectrum of [2] in DMSO-$d_6$ shows relatively downfield absorptions for all the protons due to coordination of ligand lone pairs with Mo cations (Table 7). Two equivalent C–H protons of imidazole appear as singlets, because of the tautomerization equilibrium.

**Table 7: ($^1$H NMR absorptions in ppm)**

| Assignments | Imidazole[64,65] in $CDCl_3$ | [2] |
|---|---|---|
| N-H | 11.60 1H | 14.94 (b) 2H↓ |
| C-H (in middle of nitrogen atoms) | 7.72 1H | 9.21 (s) 1H↓ |
| C-H (remaining) | 7.14 2H | 7.74 (s) 2H↓ |
| Residual[63] DMSO-$d_6$ | | 2.58 (s) |
| THF[63] C-2, 5 | | 3.63 (s) 4H |
| THF[63] C-3, 4 | | |

On comparison of $^1$H NMR of 3-methylpyridine[58,59,66] with that of [3], it is observed that these absorptions have moved downfield due to decrease in π-electron density of the ring on lone pair sharing by nitrogen with molybdenum cation (Table 8).

**Table 8: ($^1$H NMR absorptions in ppm)**

| Absorptions | 3-Methylpyridine[58,59,66] in $CDCl_3$ | [3] |
|---|---|---|
| H ($CH_3$) | 2.32 3H Singlet | 2.57↓ |
| H-$C_1$ | 8.44 1H Singlet | 8.89 1H ↓ |
| H-$C_3$ | 7.45 1H Doublet | 8.47 1H ↓ |
| H-$C_4$ | 7.16 1H Triplet | 8.01 ↓ |
| H-$C_5$ | 8.42 1H Doublet | 8.83 ↓ |
| Residual[63] DMSO-$d_6$ | | 2.58 |
| THF[63] C-2, 5 | | 3.65 |
| THF[63] C-3, 4 | | 1.56 |

On comparison of $^1$H NMR of 4-methylpyridine[58,59,66] with that of [4] it is observed that these absorptions have moved downfield due to decrease in π-electron density of the ring on lone pair sharing by nitrogen with molybdenum cation (Table 9).

**Table 9: ($^1$H NMR absorptions in ppm)**

| Assignments | 4-Methylpyridine[58,59,66] in $CDCl_3$ | [4] |
|---|---|---|
| H ($CH_3$) | 2.34 3H s | 2.58 ↓ |
| H-$C_1$ & H-$C_5$ | 8.46 2H d | 8.78 ↓ |
| H-$C_2$ & H-$C_4$ | 7.10 2H d | 7.88 ↓ |
| Residual[63] DMSO-$d_6$ | | 2.50 |
| THF[63] C-2, 5 | | 3.32 |
| THF[63] C-3, 4 | | 1.49 |

## $^{13}$C NMR Spectra

On comparison of $^{13}$C NMR of succinimide[67] with that of [1], it is observed that these absorptions have moved slightly upfield (Table 10).

**Table 10: ($^{13}$C NMR absorptions in ppm)**

| Assignments | Succinimide[67] | [1] |
|---|---|---|
| C attached to oxygen | 183.85 | 179.30 singlet |
| Other C | 30.30 | 29.41 singlet |
| Residual[68] DMSO-$d_6$ | | 39.37 pentet |
| THF[69] C-2, 5 | | |
| THF[69] C-3, 4 | | |

## Microbiological activity

Molybdenum compounds prepared were tested for their antibacterial and antifungal activities with strains: *Gram-positive* bacteria, *Staphylococcus aureus* (MTCC-737), *Gram-negative* bacteria, *E. coli* (MTCC-1687), fungi *Candida albicans* (MTCC-227) and *Aspergillus niger* (MTCC-282). Reference drugs amoxicillin and ketoconazole were used for bacteria and fungi, respectively. Zone of inhibition[70] for a strain of bacteria/fungi has been measured to find out extent of resistance of bacteria/fungi to the reference drug. Molybdenum compounds have been observed potentially active against bacteria and fungi (Table 11). Especially,

1. Compounds 1,2,3 and 4 have greater antibacterial potential against *E. coli* than the reference drug (amoxicillin).
2. Compounds 3 and 4 have greater antifungal potential against *C. albicans* than the reference drug (ketoconazole).

## Mass Spectra (LC-MS)[71]

Ionic species noted (Tables 12,13) justify the formulae,

**Table 11: (Microbiological Study)**

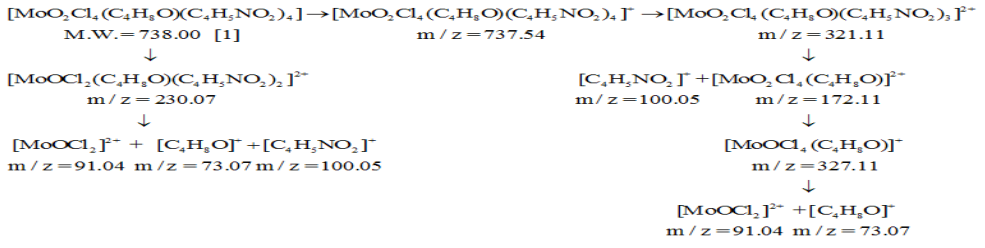
| Compound (100 μg/mL) | Zone of inhibition[70] (mm) | | | |
|---|---|---|---|---|
| | *Gram-positive* | *Gram-negative* | Antifungal | |
| | *S. aureus* | *E. coli* | *C. albicans* | *A. niger* |
| Reference Drug | 25.69 | 18.35 | 21.37 | 28.21 |
| [1] | 21.38 | 19.51 | 18.54 | 21.29 |
| [2] | 18.63 | 21.41 | 19.66 | 22.51 |
| [3] | 21.41 | 21.36 | 22.57 | 19.12 |
| [4] | 19.36 | 21.58 | 22.32 | 18.74 |
| Solvent control | --- | --- | --- | --- |

Conclusion and results colon: These compounds can kill and inhibit the growth of microbes

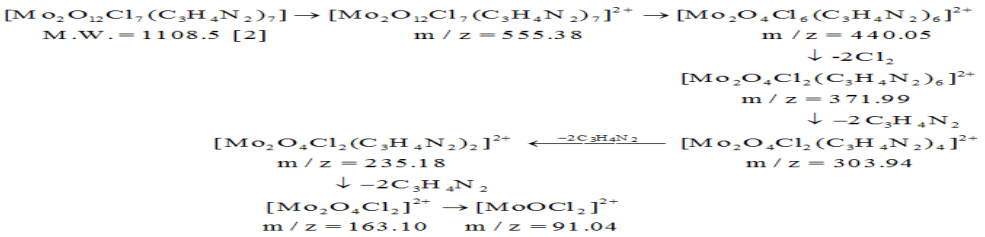
**Table 12: (LC-MS Ionization)**

Compounds

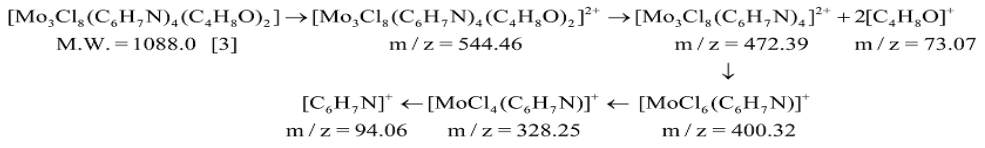
[1]




[2]




[3]




[4]

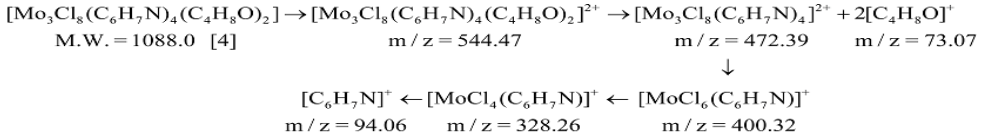



**Table 13: (LC-MS Ion m/z values)**

| Compounds | Ion | Calculated[71] | Detected | Relative intensity |
|---|---|---|---|---|
| [1] | $[MoO_2Cl_4(C_4H_8O)(C_4H_5NO_2)_3]^{2+}$ | 319.46 | 321.11 | 15% |
| | $[MoO_2Cl_4(C_4H_8O)(C_4H_5NO_2)_4]^{+}$ | 737.95 | 737.54 | 2% |
| | $[MoOCl_4(C_4H_8O)]^{+}$ | 325.83 | 327.11 | 42% |
| | $[MoOCl_2(C_4H_5NO_2)_2(C_4H_8O)]^{2+}$ | 226.97 | 230.07 | 36% |
| | $[C_4H_5NO_2]^{+}$ | 99.03 | 100.05 | 52% |
| | $[C_4H_8O]^{+}$ | 72.05 | 73.07 | 13% |
| | $[MoOCl_2]^{2+}$ | 91.91 | 91.04 | 100% |
| | $[MoO_2Cl_4(C_4H_8O)]^{2+}$ | 170.91 | 172.11 | 8% |
| [2] | $[Mo_2O_{12}Cl_7(C_3H_4N_2)_7]^{2+}$ | 554.40 | 555.38 | 4% |
| | $[Mo_2O_4Cl_6(C_3H_4N_2)_6]^{2+}$ | 438.91 | 440.05 | 7% |
| | $[Mo_2O_4Cl_2(C_3H_4N_2)_6]^{2+}$ | 368.97 | 371.99 | 12% |
| | $[Mo_2O_4Cl_2(C_3H_4N_2)_4]^{2+}$ | 300.93 | 303.94 | 3% |
| | $[Mo_2O_4Cl_2(C_3H_4N_2)_2]^{2+}$ | 232.90 | 235.18 | 2% |
| | $[Mo_2O_4Cl_2]^{2+}$ | 164.86 | 163.10 | 8% |
| | $[MoOCl_2]^{2+}$ | 91.91 | 91.04 | 10% |
| | $[C_3H_4N_2]^{+}$ | 68.03 | 69.04 | 100% |
| [3] | $[Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2]^{2+}$ | 544.90 | 544.46 | 3% |
| | $[Mo_3Cl_8(C_6H_7N)_4]^{2+}$ | 472.84 | 472.39 | 4% |
| | $[MoCl_6)(C_6H_7N)]^{+}$ | 400.77 | 400.32 | 5% |
| | $[MoCl_4)(C_6H_7N)]^{+}$ | 330.83 | 328.25 | 10% |
| | $[C_6H_7N]^{+}$ | 93.05 | 94.06 | 100% |
| | $[C_4H_8O]^{+}$ | 72.05 | 73.07 | 8% |
| [4] | $[Mo_3Cl_8(C_6H_7N)_4(C_4H_8O)_2]^{2+}$ | 544.90 | 544.47 | 6% |
| | $[Mo_3Cl_8(C_6H_7N)_4]^{2+}$ | 472.84 | 472.39 | 8% |
| | $[MoCl_6)(C_6H_7N)]^{+}$ | 400.77 | 400.32 | 18% |
| | $[MoCl_4)(C_6H_7N)]^{+}$ | 330.83 | 328.26 | 18% |
| | $[C_6H_7N]^{+}$ | 93.05 | 94.06 | 100% |
| | $[C_4H_8O]^{+}$ | 72.05 | 73.07 | 3% |

## CONCLUSION

Band at 3434 $cm^{-1}$ suggests the presence of N-H group in [1]. Stretching at 938 $cm^{-1}$ and 918 $cm^{-1}$ reveal the existence of cis-$MoO_2^{2+}$ core in [1]. C=O frequency has not altered much from 1773 $cm^{-1}$ and 1711 $cm^{-1}$, indicating thereby absence of Mo-O coordination in [1]. There seems to be little decrease in C=O bond order. $^1H$ NMR of [1] shows $CH_2$ peaks at 2.63 ppm showing upfield shift. $^{13}C$ NMR of [1] shows that the absorptions have moved slightly upfield. Compound is potentially active against microbes. Detection of ions in LC-MS are in tune with the formula presented.

There is presence of broad band at 3431 $cm^{-1}$-3000 $cm^{-1}$ pertaining to N-H group in [2]. This broadening occurs in the solid state (KBr disk) because of hydrogen bonding. A strong band at 975 $cm^{-1}$ is suggestive of terminal Mo=O in [2]. Imidazole spectrum shows C-H proton (in middle of nitrogen atoms) absorption at 7.72 ppm. Remaining C-H protons absorb at 7.14 ppm. N-H absorption occurs at 11.60 ppm. Spectrum of [2] shows relatively downfield absorptions for all the protons due to coordination of ligand lone pairs with Mo cations. Two equivalent C-H protons of imidazole appear as singlets because of the tautomerization equilibrium. Compound is potentially active against microbes. Detection of ions in LC-MS are in tune with the formula presented.

Band at 3055 $cm^{-1}$ has been located in [3]. There is increase of ring C=N str. & ring C=N torsion values and decrease of ring C-H bending mode values, which show presence of some Mo(d$\pi$)$\rightarrow$N(p$\pi$) back bonding. A strong band at 977 $cm^{-1}$ is suggestive of terminal Mo=O in [3]. It is observed that these proton absorptions have moved downfield due to decrease in $\pi$-electron density of the ring on lone pair coordination by nitrogen with molybdenum cation. Compound is potentially active against microbes. Detection of ions in LC-MS are in tune with the formula presented.

Band at 3097 $cm^{-1}$ has been located in [4]. There is increase of ring C=N str. & ring C=N torsion values and decrease of ring C-H bending mode values, which show presence of some Mo(d$\pi$)$\rightarrow$N(p$\pi$) back bonding. A strong band at 976 $cm^{-1}$ is suggestive of terminal Mo=O in [4]. It is observed that these proton absorptions have moved downfield due to decrease in $\pi$-electron density of the ring on lone pair coordination by nitrogen with molybdenum cation. Compound is potentially active against microbes. Detection of ions in LC-MS are in tune with the formula presented.

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**Conflict of interest**

Authors have no conflict of interest.

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