



Reactions of MoOCl_4 , MoO_2Cl_2 & MoCl_5 with Potassium phthalimide, Imidazole, Pyrazole, Succinimide, 2-Mercaptopyridine & 2-Mercaptopyridine-N-oxide sodium

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Abstract: MoOCl_4 reacts with pot phthalimide, 2-mercaptopyridine, 2-mercaptopyridine-N-oxide sodium in CH_3CN medium to yield multinuclear Mo complexes: $\text{Mo}_2\text{O}_3\text{Cl}(\text{CH}_3\text{CN})_2(\text{C}_8\text{H}_5\text{O}_2\text{N})_2\text{Cl}_7$ [1], $\text{Mo}_2\text{O}_3\text{Cl}(\text{CH}_3\text{CN})_2(\text{C}_8\text{H}_5\text{O}_2\text{N})_4\text{Cl}_5$ [2], $\text{Mo}_2\text{O}_4\text{Cl}_4(\text{C}_5\text{H}_4\text{NS-SNC}_5\text{H}_4)$ [8], $\text{MoOCl}_3(\text{C}_5\text{H}_4\text{NS-SNC}_5\text{H}_4)$ [9] & $\text{Mo}_2\text{O}_4\text{Cl}_4(\text{C}_5\text{H}_4\text{NS-SNC}_5\text{H}_4)$ [10], MoOCl_4 reacts with succinimide in CH_2Cl_2 to form $\text{MoO}_2\text{Cl}_2(\text{C}_4\text{H}_5\text{O}_2\text{N})$ [11]. MoO_2Cl_2 reacts with pot phthalimide & succinimide in CH_3CN to provide $\text{MoO}_2(\text{C}_8\text{H}_5\text{O}_2\text{N})_2$ [4] & $\text{MoO}_2\text{Cl}_2(\text{C}_4\text{H}_5\text{O}_2\text{N})_2$ [5], respectively. MoCl_5 reacts with pot phthalimide, pyrazole & imidazole in CH_3CN to yield $\text{MoO}_2\text{Cl}_2(\text{CH}_3\text{CN})(\text{C}_8\text{H}_5\text{O}_2\text{N})\text{Cl}_2$ [3], $\text{MoO}_2\text{Cl}_2(\text{C}_3\text{H}_4\text{N}_2)$ [6] & $\text{MoO}_2\text{Cl}_2(\text{C}_3\text{H}_4\text{N}_2)\text{Cl}$ [7], resp. Compounds have been characterized by elemental analysis, ^1H NMR, FTIR, Mass (GC-MS), electronic spectra and molar conductance studies.

Keywords: MoOCl_4 ; MoO_2Cl_2 ; MoCl_5 ; pot phthalimide; succinimide; pyrazole; imidazole; 2-mercaptopyridine; 2-mercaptopyridine-N-oxide sodium.

I. Introduction

It is already known that Molybdenum forms multinuclear complexes.^{1, 2, 3, 4, 5, 6} These complexes may be cyanide bridged^{1, 2} when CH_3CN medium is used as reaction medium. CH_3CN may act as terminal or cross bridged ligand. Identification of terminal^{4, 5, 7, 8}/cross bridged^{1, 4, 5, 6} CH_3CN ligand can be done by FTIR studies. Cross-bridging¹ of CH_3CN ligand can also be identified by ^1H NMR chemical shifts.

The author reported^{9, 10, 11, 12, 13} reactions of MoOCl_4 , MoO_2Cl_2 , MoCl_5 in CH_2Cl_2 medium. Owing to feeble solubility of MoO_2Cl_2 , MoCl_5 and ligands in CH_2Cl_2 medium, reactions of MoOCl_4 & MoO_2Cl_2 & MoCl_5 were carried out and reported^{4, 5} by the author in CH_3CN medium. In reactions reported in this paper, enhanced polarity of CH_3CN medium has played an important role in reaction process. Products obtained are mostly soluble in CH_3CN . This was due to better solubility of MoOCl_4 , MoO_2Cl_2 & MoCl_5 in CH_3CN due to formation of solvent stabilized complexes, like $\text{MoO}_2\text{Cl}_2(\text{CH}_3\text{CN})_2$.

Many compounds reported in this paper are multinuclear as assessed by the fragments detected in mass (GC-MS) spectra. There is some similarity in the fragments formed from the compounds prepared from MoOCl_4 , MoO_2Cl_2 & MoCl_5 .

Phthalimide^{14, 15} derivatives have been reported with biological activities. Among these activities, like inhibition of tumor necrosis factor- α .

Succinimides¹⁶ are important class of substrates for biological applications.

Pyrazoles and their derivatives exhibit various biological activities including antimicrobial¹⁷, anti-cyclooxygenase¹⁸, anticonvulsant¹⁹, antitubercular²⁰, antitumor²¹, antiinflammatory²², analgesic²³, antidiabetic³⁴, antipshycotic^{25, 26, 27}. Many drugs, such as, antifungal drugs and nitroimidazole contain an imidazole^{28, 29} ring.

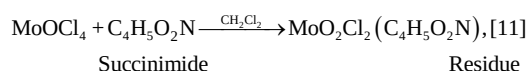
Imidazole³⁰ containing drugs have a broad scope in remedying various dispositions in clinical medicine.

Compounds containing N-oxides and Sulphur, such as 2-mercaptopyridine and 2-mercaptopyridine-N-oxide, possess important biological activity^{31, 32}. Compounds containing sulphur, as well as their metal complexes exhibit numerous biochemical applications^{33, 34, 35}.

II. Experimental

Materials:

Molybdenumoxytetrachloride MoOCl_4 has been prepared in laboratory by reaction of MoO_3 (CDH, AR Grade) with SOCl_2 at reflux temperature for about 5 h. After refluxing, excess SOCl_2 was evacuated in liquid nitrogen traps. Dark green crystals thus obtained were dissolved in dry CH_2Cl_2 to get a dark red solution. Solution was filtered through filtration unit fitted with G-4 sintered glass crucible to remove any unreacted/undissolved MoO_3 .



III. Results & Discussions

Analytical Measurements:

All compounds are very moisture and air sensitive. They are insoluble in common organic solvents like CH_2Cl_2 , CHCl_3 , n-hexane, but soluble in polar solvents like CH_3CN , DMSO and DMF. Formulations of these compounds have been done on basis of their elemental analytical data, mass (GC-MS) and molar conductance Λ_M measurements. Analytical data and mass (GC-MS) spectra reveal that some of these compounds are multinuclear. Λ_M of compounds [1] to [11] in DMSO are $0.1 - 1.9 \text{ cm}^2\text{ohm}^{-1}\text{mol}^{-1}$, suggesting their non-electrolytic nature (Table-1).

Table-1 (Analytical & Molar Conductance Data)								
Compounds (Colour/F.W.)	Λ_M *	% Composition Observed (Calculated)						
		Mo	Cl	C	H	N	O	S
$\text{Mo}_2\text{O}_3\text{Cl}(\text{CH}_3\text{CN})_2(\text{C}_8\text{H}_5\text{O}_2\text{N})_2\text{Cl}_7$, [1] (Light green/900.0)	0.6	20.85 (21.33)	30.70 (31.55)	25.86 (26.66)	1.84 (1.77)	05.39 (06.22)	11.7 (12.4)	----
$\text{Mo}_2\text{O}_3\text{Cl}(\text{CH}_3\text{CN})_2(\text{C}_8\text{H}_5\text{O}_2\text{N})_4\text{Cl}_5$, [2] (Navy blue/1123.0)	0.4	17.97 (17.09)	18.05 (18.96)	37.65 (38.46)	2.57 (2.31)	06.50 (07.47)	16.4 (15.6)	----
$\text{MoO}_2\text{Cl}_2(\text{CH}_3\text{CN})(\text{C}_8\text{H}_5\text{O}_2\text{N})\text{Cl}_2$, [3] (Light blue/458.0)	0.2	20.00 (20.96)	30.22 (31.00)	25.72 (26.20)	2.24 (1.74)	05.83 (06.11)	13.1 (13.9)	----
$\text{MoO}_2(\text{C}_8\text{H}_4\text{O}_2\text{N})_2$, [4] (White/420.0)	0.9	21.95 (22.85)	----	44.98 (45.71)	2.52 (1.90)	05.80 (06.66)	22.1 (22.8)	----
$\text{MoO}_2\text{Cl}_2(\text{C}_4\text{H}_5\text{O}_2\text{N})_2$, [5] (White/397.0)	0.1	23.54 (24.18)	17.04 (17.88)	23.26 (24.18)	3.40 (2.51)	06.98 (07.05)	23.6 (24.1)	----
$\text{MoO}_2\text{Cl}_2(\text{C}_3\text{H}_4\text{N}_2)$, [6] (Dark blue/267.0)	0.1	35.04 (35.95)	25.73 (26.59)	12.86 (13.48)	2.28 (1.49)	09.55 (10.48)	11.1 (11.9)	----
$\text{MoO}_2\text{Cl}_2(\text{C}_3\text{H}_4\text{N}_2)\text{Cl}$, [7] (Dark brown/302.5)	0.2	30.85 (31.73)	34.67 (35.20)	12.73 (11.90)	2.14 (1.32)	10.12 (09.25)	09.8 (10.5)	----
$\text{Mo}_2\text{O}_4\text{Cl}_4(\text{C}_5\text{H}_4\text{NS-SNC}_5\text{H}_4)$, [8] (Dark brown/618.0)	0.4	30.14 (30.96)	22.13 (22.90)	18.78 (19.35)	2.18 (1.61)	04.32 (04.51)	09.6 (10.3)	09.6 (10.3)
$\text{MoOCl}_3(\text{C}_5\text{H}_4\text{NS-SNC}_5\text{H}_4)$, [9] (Dark brown/438.5)	1.9	20.99 (21.89)	23.87 (24.38)	26.78 (27.36)	2.43 (1.82)	05.63 (06.38)	02.9 (03.6)	13.7 (14.5)
$\text{Mo}_2\text{O}_4\text{Cl}_4(\text{C}_5\text{H}_4\text{NS-SNC}_5\text{H}_4)$, [10] (Light brown/618.0)	0.9	30.23 (30.96)	21.99 (22.90)	18.55 (19.35)	2.21 (1.61)	05.22 (04.51)	09.9 (10.3)	09.74 (10.3)
$\text{MoO}_2\text{Cl}_2(\text{C}_4\text{H}_5\text{O}_2\text{N})$, [11] (Light green/298.0)	0.6	31.84 (32.21)	22.98 (23.82)	17.07 (16.10)	2.62 (1.67)	04.50 (04.69)	20.8 (21.4)	----

* Molar conductance of millimolar solutions in DMSO at 25°C .

FTIR Spectra:

Potassium phthalimide³⁷ shows N-H stretchings at 3269 cm^{-1} and 3154 cm^{-1} . Band at 3205.14 cm^{-1} shows that N-H group is present in compound [1]. Strong band at 911.17 cm^{-1} show the presence of terminal $\text{Mo}=\text{O}$ group^{22, 23} in the compound [1] (Table-2).

Band at 3201.1 cm^{-1} shows that N-H group is present in compound [2]. Strong band at 956.2 cm^{-1} show the presence of terminal $\text{Mo}=\text{O}$ group^{38, 39} in the compound [2] (Table-2).

Band at 3204.0 cm^{-1} shows that N-H group is present in compound [3]. Strong bands at 992.1 s cm^{-1} and 906.1 w cm^{-1} show the presence of cis- MoO_2^{2+} core⁴⁰ in the compound [3] (Table-3).

Band at 3199.2 cm^{-1} shows that N-H group is present in compound [4]. Strong bands at 950.6 cm^{-1} and 906.3 s cm^{-1} show the presence of cis- MoO_2^{2+} core⁴⁰ in the compound [4] (Table-3).

Succinimide^{41, 42} shows N-H stretchings at 3410 cm^{-1} & 3223 cm^{-1} . Bands at 3478.13 cm^{-1} and 3199.2 cm^{-1} show that N-H group is present in compound [5]. Strong bands at 977.3 cm^{-1} & 908 cm^{-1} show the presence of cis- MoO_2^{2+} core⁴⁰. There is no shift in $\text{C}=\text{O}$ stretching frequency from 1771 cm^{-1} , which indicates that there is no $\text{Mo}-\text{O}$ coordination bond in compound [5] (Table-4).

Pyrazole^{43, 44} shows N-H stretching at 3450 cm^{-1} . Band at 3278.2 cm^{-1} shows that N-H group is present in compound [6]. $\text{Mo}-\text{N}$ coordination bond lowers frequency of N-H stretching. Strong bands at 981.4 s cm^{-1} and 917.6 cm^{-1} show the presence of cis- MoO_2^{2+} core⁴⁰ in compound [6] (Table-5).

Imidazole^{45, 46} shows N-H stretchings at 3724-3237 cm⁻¹. Band at 3278.1 cm⁻¹ shows that N-H group is present in compound [7]. This peak is broad in the solid state (KBr disk) due to hydrogen bonding. Two bands at 990.3 cm⁻¹ and 913.5 cm⁻¹ are attributable to the presence of cis-MoO₂²⁺ core⁴⁰ in compound [7] (Table-6). 2-Mercaptopyridine^{47, 48, 49, 50, 51} shows N-H stretchings at 3338.21 cm⁻¹, 3209.2 cm⁻¹ and S-H str at 2709 cm⁻¹. Bands at 3338.21 cm⁻¹, 3209.2 cm⁻¹ and 3141.2 cm⁻¹ show that N-H group may be present in compound [8]. Absence of any band in the region of 2709 cm⁻¹ shows absence of S-H group in compound [8] (Table-7). Bands above 3100 cm⁻¹ are missing which shows that N-H group is missing in compound [9]. Absence of any band in the region of 2709 cm⁻¹ shows absence of S-H group in compound [9] (Table-7). 2-Mercaptopyridine-N-oxide sodium⁵² shows N-H stretching above 3100 cm⁻¹. Band at 3392.36 cm⁻¹ shows that N-H group may be present in compound [10]. Absence of any band in the region of 2709 cm⁻¹ shows absence of S-H group in compound [10] (Table-7).

Table 2 (FTIR absorptions in cm ⁻¹)			
Assignments	C ₈ H ₄ O ₂ NK ⁺ (Pot. Phthalimide) ³⁷	[1]	[2]
N-H str	3269 w, 3154 w	3205.14 s, b	3201.1 s, b
In phase C=O str	1704 m, 1687 w, 1665 vvw	1744.10 s, 1641.21 m	1734.0 s, b, 1643.0 s
C-C Benzene ring str	1608 s	1604.21 m	1603.1 s
Out of phase C=O str	1593 s, 1588 s, 1552 vw, 1507 vw	-----	1555.0 s, b
C-C Benzene ring str	1465 vvw, 1449 vvw, 1398 w, 1372 s, 1356 m	1469.32 vw, 1405.24 m, 1387.24 m	1466.2 sh, 1389.0 s, b
Five membered ring C-C str	1297 s, 1231 w, 1209 vw, 1184 s, 1128 s	1307.26 m, 1259.22 m	1304.1 s, 1258.1 s, 1171.7 sh, 1141.7 w
C-H in plane bending	1091 vvw	1053.30 w	1073.5 w, 1052.3 s
C-C Benzene ring str	769 vw, 756 vw, 743 vw	746.22 m	745.1 sh
C-H out of plane bending	717 s	714.11 s	714.0 s
Mo=O Str. ^{38, 39}	----	911.17 s	956.2 s

Table 3 (FTIR absorptions in cm ⁻¹)			
Assignments	C ₈ H ₄ O ₂ NK ⁺ (Pot. Phthalimide) ³⁷	[3]	[4]
N-H str	3269 w, 3154 w	3204.0 s, b	3199.2 s, b
In phase C=O str	1704 m, 1687 w, 1665 vvw	1730.0 s, b, 1698.0 s, b	1731.0 s, b, 1647.4 s
C-C Benzene ring str	1608 s	1604.3 sh	1600.5 s
Out of phase C=O str	1593 s, 1588 s, 1552 vw, 1507 vw	----	1580.4 s
C-C Benzene ring str	1465 vvw, 1449 vvw, 1398 w, 1372 s, 1356 m	1406.1 s, 1389.2 sh	1403.1 s, 1387.4 m
Five membered ring C-C str	1297 s, 1231 w, 1209 vw, 1184 s, 1128 s	1307.2 s, 1282.2 s	1306.5 s, 1259.6 s
C-H in plane bending	1091 vvw	1071.8 w, 1052.5 m	1073.2 w, 1052.14 m
C-C Benzene ring str	769 vw, 756 vw, 743 vw	796.8 m, 738.3 m	795.5 m, 740.5 m
C-H out of plane bending	717 s	715.1 s	714.1 s
Mo=O Str. of cis-MoO ₂ ²⁺ core ⁴⁰	----	992.1 s, 906.1 w	950.6 m, 906.3 s

Table 4 (FTIR absorptions in cm ⁻¹)			
Assignments	C ₄ H ₅ NO ₂ (Succinimide) ^{41, 42}	[5]	[11]
NH str.	3410 s, b, 3223 s, b	3478.13 sh, 3199.2 s	3510.9 sh, 3300.5 s
CO sym., HNC in plane bending	1771 m	1769.3 m	1775.19 m, 1756.19 m,
CO asym., HNC in plane bending	1713 vs, b	1697.2 s	1682.0 s, 1595.13 sh
CH ₂ sym. scissoring	1431 m	1424.8 m	1416.26 sh
CH ₂ asym. scissoring	1400 m	1366.7 m	1373.15 m
CNC asym. str., HNC in plane bending	1349 s, 1334	1315.7 m	1297.17 m
CH ₂ bending, ring in plane bending	1294 s	1287.3 m	1297.17 m
CH ₂ bending	1241	11247.12 m	1248.28 w
CNC asym. str., HNC in plane bending	1186 s,	1177.9 m	1182.4 s
C-C str., CNC sym. str.	849	848.5 w	858.3 w
CH ₂ bending, ring out of plane	817 s	818.2 m	815.17 m, 721.13 s

bending			
OCN asym. out of plane bending	632 m	637.2 m	646.15 m
OCN sym. out of plane bending, CH ₂ bending	543 w	547.2 w	553.29 w
Mo=O Str. of cis-MoO ₂ ²⁺ core ⁴⁰	----	977.3 s, 908 m	981.10 s, 919.19 m

Table 5 (FTIR absorptions in cm ⁻¹)		
Assignments	C ₃ H ₄ N ₂ (Pyrazole) ^{43, 44}	[6]
N-H str.	3450	3278.2 vs, vb
C-H str.	3155, 3144	3138.1 vs, b
C=C ring str.	1558	1615.4 s, 1592.7 sh
N-C ring str.	1468, 1138	1464.11 sh, 1409.8 m, 1323.14 sh, sh, 1260.15 sh,, 1167.16 sh
C-H in plane bending	1046, 1035	1111.9 s, 1052.9 s
C-H bending	938	----
Ring bending	918	----
C-H out of plane bending (wagging), Ring twisting	893, 840, 760	784.1 s, b
Ring twisting	656, 619	635.7 sh, 608.7 m, 565.4 m
Ring twisting, N-H wagging	498	433.16 w
Mo=O Str. of cis-MoO ₂ ²⁺ core ⁴⁰	----	981.4 s, 917.6 m

Table 6 (FTIR absorptions in cm ⁻¹)		
Assignments	C ₃ H ₄ N ₂ (Imidazole) ^{45, 46}	[7]
N-H str.	3724 vb, 3656 vb, 3270, 3241, 3237	3278.1 s, br
C-H str.	3196, 3165	3142.1 s, 2984.4 sh, 2848.6 sh,
C=C ring str.	1558, 1500	1608.7 s, 1581.5 s
N-C ring str.	1434	1419.11 m
C-H in plane bending	1092, 1074	1089.12 m, 1045.8 s
C-H bending	964	----
Ring bending	909	----
C-H out of plane bending (wagging), Ring twisting	816, 730	884.5 s, 764.2 s, br
Ring twisting	646	620.4 s
Ring twisting, N-H wagging	528	509.17 m
Mo=O str. of cis-MoO ₂ ²⁺ core ⁴⁰	----	990.3 s, 913.5 m

Table 7 (FTIR absorptions in cm ⁻¹)				
Assignments	C ₅ H ₅ NS ^{37, 38, 39, 40, 41, 42} (2-Mercaptopyridine)	[8]	[9]	[10]
N-H str	3176	3338.21 vs, b, 3209.2 s, 3141.2 s	---	3392.36s, b,
C-H str	3054, 2927, 2879	3025.21 s, 2891.23 m	3077.21 m, 2929.25 m	3101.41 w
S-H str	2709 m	---	---	---
Ring breathing modes & Hydrogen in plane wagging	1613 s, 1576 vs, 1504 s, 1446 s, 1418 s	1601.14 s, 1526.24 w, 1509.25 w, 1446.21 m, 1378.26 m	1603.17 s, 1581.16 s, 1506.26 m, 1494.26 m	1607.41 s, 1528.54 w, 1464.47 w, 1428.48 w
C=N ring	1275 m	1274.28 m	1283.26 m	1277.53 w
C-H in plane bending	1246 m	1225.34 w	1219.33 w	1246.56 vw
C-S str	1188 vs, 1145 vs	1167.32 w	1145.1 w	1199.46 m
C=S str, Hydrogen out of plane bendingout of plane	745, 613	760.10 vs, 621.25 sh,	772.1 vs, 711.3 w, 622.37 w	770.31 vs, 702.42 sh
Mo=O str. of cis-MoO ₂ ²⁺ core ⁴⁰	----	977.16 vs, 915.28 w	---	944.43 m, 908.35 s
Mo=O ^{38, 39} str.	----	---	983.16 vs	

¹H NMR Spectra:

Spectrum of pot. phthalimide^{53, 54} shows peaks in the range 7.3 ppm-7.8 ppm due to ring protons. Spectrum of [1] in DMSO-d₆ shows peaks due to ring protons in the range 7.03 ppm-7.98 ppm with highest peak at 7.60 ppm, N-H peak at 11.19 ppm and CH₃CN peak at 1.97 ppm. This shows presence N-H group and CH₃CN in the compound [1] (Table-8).

Spectrum of [2] in DMSO- d_6 shows peaks due to ring protons in the range 7.21 ppm-8.01 ppm with highest peak at 7.74 ppm, N-H peak at 11.26 ppm and CH_3CN peak at 1.97 ppm. This shows presence N-H group and CH_3CN in the compound [2] (Table-8).

Spectrum of [3] in DMSO- d_6 shows peaks due to ring protons in the range 7.20 ppm-8.19 ppm with highest peak at 7.74 ppm, N-H peak at 11.24 ppm and CH_3CN peak at 1.97 ppm. This shows presence N-H group and CH_3CN in the compound [3] (Table-9).

Spectrum of [4] in DMSO- d_6 shows peaks due to ring protons in the range 7.08 ppm-7.75 ppm with highest peak at 7.73 ppm, N-H peak at 11.24 ppm. This shows presence N-H group in compound [4] (Table-9).

Spectrum of succinimide^{55, 56} shows CH_2 peaks at 2.73 ppm. Spectrum of compound [5] in DMSO- d_6 shows peak that peak due to CH_2 has moved up field (Table-10).

Spectrum of pyrazole⁴¹ in CCl_4 shows absorptions due to middle CH proton at 6.31 ppm, CH protons on side two carbons at 7.61 ppm and due to N-H proton at 12.64 ppm. Spectrum of compound [6] in DMSO- d_6 shows peak that peak due to middle CH proton and on side two carbons have moved downfield (Table-11). Peaks of C-H protons of pyrazole appear as singlets, because of the tautomerization equilibrium.

Spectrum of imidazole⁵⁸ in $CDCl_3$ shows absorptions due to CH proton (between two nitrogen atoms) at 7.73 ppm, CH protons on other two carbons at 7.15 ppm and due to N-H proton at 11.62 ppm. Spectrum of compound [7] in DMSO- d_6 shows relatively up field absorptions for CH proton (between two nitrogen atoms), CH protons on other two carbons (Table-12). Two equivalent C-H protons of imidazole appear as singlets, because of the tautomerization equilibrium.

Spectrum of 2-mercaptopyridine⁵⁹ shows peaks at 7.33 ppm, 6.80 ppm, 7.46 ppm and 7.70 ppm due to C3-H, C4-H, C5-H and N-C-H protons, respectively. Spectra of compounds [8], [9] and [10] show that absorptions move downfield (Table-13). N-H peaks were not observed.

Spectrum of compound [11] in DMSO- d_6 shows peak that peak due to CH_2 has moved up field (Table-10).

Table 8 (^1H NMR absorptions in ppm)			
Assignments	$C_8H_4O_2N^+K^+$ (Pot. phthalimide) ^{53, 54}	[1]	[2]
N-H	----	11.19 (s) 1H	11.26 (s) 1H
Ring C-H	7.58 -7.87	7.03 -7.98 (m) 10H	7.21 -8.01 (m) 20H
CH_3CN	----	1.97 (s) 6H	1.99 (s) 6H

Table 9 (^1H NMR absorptions in ppm)			
Assignments	$C_8H_4O_2N^+K^+$ (Pot. phthalimide) ^{53, 54}	[3]	[4]
N-H	----	11.24 (s) 1H	11.23 (s) 1H
Ring C-H	7.58 -7.87	7.20 -8.19 (m) 5H	7.08 -7.75 (m) 20H
CH_3CN	----	1.97 3H	----

Table-10 (^1H NMR absorptions in ppm)			
Assignments	$C_4H_5NO_2$ (Succinimide) ^{55, 56}	[5]	[11]
N-H	----	10.96 1H	11.00 1H
CH_2	2.73	2.55 4H	2.50 4H

Table-11 (^1H NMR absorptions in ppm)		
Assignments	$C_3H_4N_2$ in CCl_4 (Pyrazole) ⁵⁷	[6]
N-H	12.64 1H	----
Middle CH_2	6.31 (s) 1H	6.45 (s) 1H
Side CH_2	7.61(s) 2H	7.92 (s) 2H

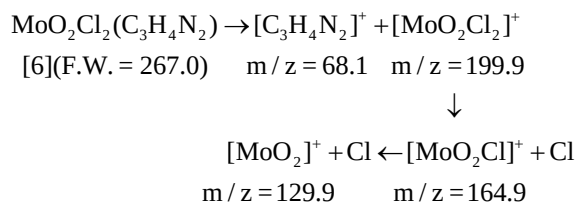
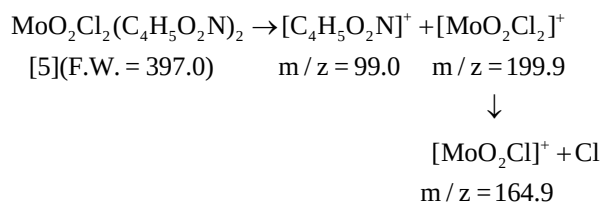
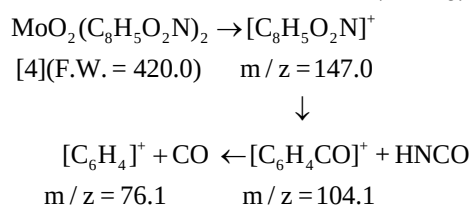
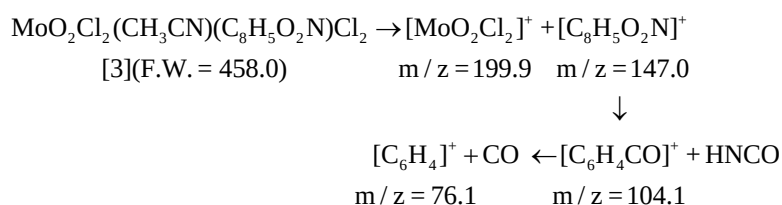
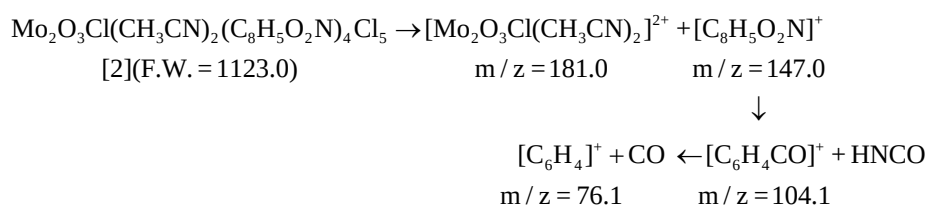
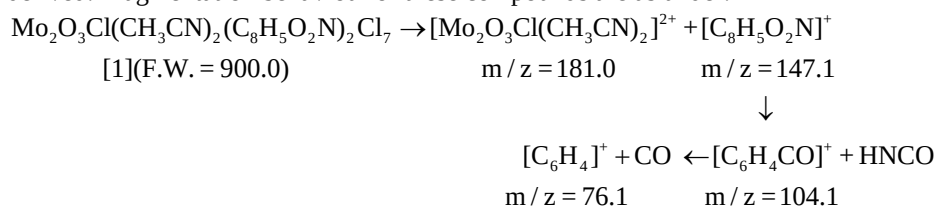
Table-12 (^1H NMR absorptions in ppm)		
Assignments	$C_3H_4N_2$ (Imidazole) ⁵⁸	[7]
N-H	12.4 1H	14.65 (s) 1H
C-H (between two nitrogens)	9.37 1H	9.11 (s) 1H
C-H (other carbons)	7.80 2H	7.64 (s) 2H

Table-13 (^1H NMR absorptions in ppm)	
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Assignments	$C_8H_5O_2NS^{59}$ (2-Mercaptopyridine)	[8]	[9]	[10]
N-H	---	---	---	---
C ₃ -H	7.33	7.70	7.64	7.48
C ₄ -H	6.80	7.34	7.28	7.36
C ₅ -H	7.46	7.86	7.81	7.71
NC-H	7.70	8.52	8.45	8.40

Mass Spectra (GC-MS)⁶⁰:

Compounds [1], [2], [8], [9], [10] and [11] have been prepared from MoOCl₄ as the precursor. Compounds [3], [6] and [7] have been prepared from MoCl₅ as the precursor. Compounds [4] and [5] have been prepared from MoO₂Cl₂ as the precursor. Mass spectra (GC-MS) of these compounds have some correlations and common fragments. For example, compounds [3], [5], [6], [7], [8] [10] and [11] decompose to form common fragment 199.9 (Table-14). Based on the fragments formed, formulae of the compounds have been derived. Fragmentation behaviour of these compounds are as under.



[8]	$[\text{MoO}_2\text{Cl}_2]^+$	199.83	199.9	100%
	$[\text{MoO}_2\text{Cl}]^+$	164.86	164.9	60%
	$[\text{MoO}_2]^+$	129.89	129.9	10%
	$[\text{Mo}]^+$	97.9	97.9	8%
[9]	$[\text{C}_5\text{H}_4\text{SN} - \text{NSC}_5\text{H}_4]^+$	220.1	220.0	100%
	$[\text{C}_5\text{H}_4\text{SN} - \text{NC}_5\text{H}_4]^+$	188.04	187.0	35%
	$[\text{C}_5\text{H}_4\text{N} - \text{NC}_5\text{H}_4]^+$	156.06	156.1	70%
	$[\text{C}_5\text{H}_4\text{N}]^+$	78.03	78.0	30%
	$[\text{C}_5\text{H}_4\text{SN}]^+$	110.00	110.0	5%
[10]	$[\text{C}_5\text{H}_4\text{SN} - \text{NSC}_5\text{H}_4]^+$	220.1	220.0	75%
	$[\text{MoO}_2\text{Cl}_2]^+$	199.83	199.9	88%
	$[\text{MoO}_2\text{Cl}]^+$	164.86	164.9	60%
	$[\text{MoO}_2]^+$	129.89	129.9	15%
	$[\text{MoO}]^+$	113.90	113.0	60%
	$[\text{C}_5\text{H}_4\text{N}]^+$	78.03	78.1	100%
[11]	$[\text{C}_4\text{H}_5\text{O}_2\text{N}]^+$	99.03	99.1	44%
	$[\text{MoO}_2\text{Cl}_2]^+$	199.83	199.9	100%
	$[\text{MoO}_2\text{Cl}]^+$	164.86	164.9	40%
	$[\text{MoO}_2]^+$	129.89	129.9	5%

Electronic Spectra:

Absorptions (λ_{max} nm) Pure Ligand	Absorptions (λ_{max} nm) Compound (solution in DMSO)
Pot phthalimide ^{65, 66, 67, 68} 200-400 nm imidazole ring $\pi \rightarrow \pi^*$	[1] 259 nm (€ 3021.4) ring $\pi \rightarrow \pi^*$
Pot phthalimide ^{65, 66, 67, 68} 200-400 nm imidazole ring $\pi \rightarrow \pi^*$	[2] 260 nm (€ 3725.9) ring $\pi \rightarrow \pi^*$
Pot phthalimide ^{65, 66, 67, 68} 200-400 nm imidazole ring $\pi \rightarrow \pi^*$	[3] 261 nm (€ 2371.2) ring $\pi \rightarrow \pi^*$
Pot phthalimide ^{65, 66, 67, 68} 200-400 nm imidazole ring $\pi \rightarrow \pi^*$	[4] 261 nm (€ 3355.0) ring $\pi \rightarrow \pi^*$
Succinimide ^{4, 5} (solution in DMSO) 259 nm	[5] 268 nm (€ 3247.8)
Pyrazole ⁶¹ (solution in DMSO) 264 nm pyrazole ring $\pi \rightarrow \pi^*$	[6] 260 nm (€ 1406.3) pyrazole ring $\pi \rightarrow \pi^*$
Imidazole ⁶² (solution in DMSO) 284 nm imidazole ring $\pi \rightarrow \pi^*$	[7] 259 nm (€ 1209.0) imidazole ring $\pi \rightarrow \pi^*$
2-Mercaptopyridine ⁶³ 343 nm ring $\pi \rightarrow \pi^*$	[8] 278 nm (€ 2373.5)
2-Mercaptopyridine ⁶³ 343 nm ring $\pi \rightarrow \pi^*$	[9] 282 nm (€ 4175.0) ring $\pi \rightarrow \pi^*$, 260 nm (€ 3439.2) ring $\pi \rightarrow \pi^*$
2-Mercaptopyridine-N-oxide sodium ^{63, 64} below 300 nm ring $\pi \rightarrow \pi^*$	[10] 273 nm (€ 6530.9) ring $\pi \rightarrow \pi^*$
Succinimide ^{4, 5} (solution in DMSO) 259 nm	[11] 260 nm (€ 3525.9)

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