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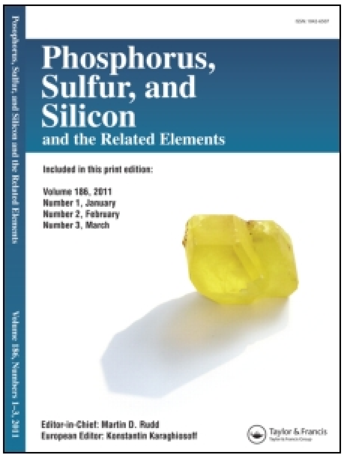

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A. A. S. El Khaldy[a]; J. R. Durig[b]
[a] Department of Chemistry, A1 Azhar University, Gaza-Palestine [b] Department of Chemistry, University of Missouri, Kansus City, Missouri, USA

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MIXED HALIDE DIALKYL AND ALKYLENEDITHIOPHOSPHATE DERIVATIVES OF RUTHENIUM (III)

A.A.S. EL KHALDY[a]* and J.R. DURIG[b]

[a]*P.O. Box 1277 Department of Chemistry. Al Azhar University, Gaza-Palestine and*
[b]*Department of Chemistry, University of Missouri-Kansas City, Missouri 302*
Scofield Hall 5100 Rockhill Road 64110–2499 U.S.A.

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Mixed chloride dialkyl and alkylenedithiophosphates of ruthenium (III), $RuCl_{3-n}[S_2P(OR)_2]n$ (R = Pr^n, and Ph) and $RuCl_{3-n}[S_2\overline{POGO}]n$ G = $-CMe_2CMe_2-$, $CH_2CMe_2CH_2-$, $-CH_2CEt_2CH_2-$, and $-CMe_2CH_2CHMe-$, n = 1, 2 have been synthesized for the first time by the reactions of ruthenium trichloride with ammonium dialkyl and alkylenedithiophosphate or alternatively by disproportionation reactions of ruthenium trichloride with ruthenium tris(dialkyl and alkylenedithiophosphates) in different stoichiometric ratios in benzene.

These new complexes have been characterized by elemental analysis, molecular weight determinations, as well as IR and NMR (^1H and ^{31}P) data. Chelated structures with bidentate dialkyl and alkylenedithiophosphates groups have been proposed for all these derivatives.

INTRODUCTION

The chemistry of ruthenium complexes with dithioligands has been well studied and is the subject of several reviews.[1–7] Yet, little was about the coordination chemistry of ruthenium with dialkyl and alkylenedithiophosphates has been reported. Although these ligands form an important class, which give stable complexes with various metals and show both monodentate[8–11] and bidentate chelation.[12–14]

The mixed halide dialkyl and alkylenedithiophosphate derivatives of ruthenium are still unknown. In view of the exciting chemistry of mixed halide derivatives of ruthenium dithiocarbomates[15], it was considered

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* Author to whom correspondence should be addressed

worthwhile to synthesize a number of tris[6] as well as mixed halide dialkyl and alkylenedithiophosphate derivatives of ruthenium(III) and investigate their chemical properties.

RESULTS AND DISCUSSION

In this communication, we report the synthesis of mixed chloride dialkyl and alkylenedithiophosphate derivatives by the reaction of ruthenium trichloride with ammonium dialkyl (eq. 1) and alkylenedithiophosphate (eq. 2) in different stoichiometric ratios and also by disproportionation reactions of metal tris(dialkyl and alkylenedithiophosphates) with ruthenium chloride(eq. 3–5).

$$\begin{aligned}&\mathrm{RuCl_{3\text{-}n} + nNH_4S_2P(OR)_2} \longrightarrow \\ &\qquad \mathrm{RuCl_{3\text{-}n}[S_2P(OR)_2]n + nNH_4Cl} \qquad (1)\\ &\mathrm{R = Pr^n\ and\ Ph.}\end{aligned}$$

$$\begin{aligned}&\mathrm{RuCl_{3\text{-}n} + nNH_4S_2\overline{POGO}} \longrightarrow \\ &\qquad \mathrm{RuCl_{3\text{-}n}[S_2\overline{POGO})_2]_n + nNH_4Cl} \qquad (2)\\ &\mathrm{G = -CMe_2CMe_2, -CH_2CMe_2CH_2-, -CH_2CEt_2CH_2-,}\\ &\mathrm{and\ -CMe_2CH_2CHMe-.}\end{aligned}$$

$$\mathrm{RuCl_3 + 2Ru[S_2P(Pr\text{-}\mathit{n})_2]_3 \longrightarrow 3\ Ru(Cl)[S_2P(Pr\text{-}\mathit{n})_2]_2} \qquad (3)$$

$$\mathrm{2RuCl_3 + Ru[S_2P(OPr\text{-}\mathit{n})_2]_3 \longrightarrow 3\ RuCl_2[S_2P(Pr\text{-}\mathit{n})_2]} \qquad (4)$$

$$\begin{aligned}&\mathrm{RuCl_3 + 2Ru[S_2\overline{POCH_2CMe_2CH_2O}]_3} \rightarrow \\ &\qquad \mathrm{3\ Ru(Cl)[S_2\overline{POCH_2CMe_2CH_2O}]_2} \qquad (5)\end{aligned}$$

All the above reactions are carried out in benzene by stirring and refluxing the reactants for about 4 hours to ensure the completion of the reactions. The products obtained have been washed by n-hexane and dried under reduced pressure. These purple or dark brown solids or sticky solid compounds (except $\mathrm{ClRu[S_2\overline{POCH_2CMe_2CH_2O}]_2}$ which is colorless and volatile liquid) are soluble in common organic solvents. Molecular weight determinations in freezing benzene exhibit their monomeric nature.

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TABLE I Synthesis & Physical properties of mixed chloro dialkyl and alkylenedithiophosphate derivatives of ruthenium(III)

Compound No.	*Reactants (g)*		*Molar ratio*	*Product Physical state*	*Yield %*	*M.P.C*	*Mol. Wt.*	*Analysis % Found (Calc.)*		
	$RuCl_3$	*Amm. Dithiophosphate*						*P*	*S*	*Cl*
1		$NH_4S_2P(OPr^n)_2$	1:1	$Cl_2Ru[S_2P(OPr^n)_2]$	88	–	376.3	7.88	16.53	18.25
	0.49	0.55		Dark brown sticky solid			(384.9)	(8.02)	(16.62)	(18.44)
2		$NH_4S_2P(OPr^n)_2$	1:2	$ClRu[S_2P(OPr^n)_2]_2$	86	–	542.4	10.54	22.56	6.22
	0.29	0.64		Brown sticky solid			(562.3)	(10.91)	(22.76)	(6.31)
3		$NH_4S_2P(OPh)_2$	1:1	$Cl_2Ru[S_2P(OPh)_2]$	81	–	456.6	6.73	14.45	15.32
	0.35	0.50		Brown sticky solid			(452.97)	(6.82)	(14.12)	(15.67)
4		$NH_4S_2P(OPh)_2$	1:2	$ClRu[S_2P(OPh)_2]_2$	80	–	687.62	8.61	18.13	4.78
	0.36	1.03		Brown solid			(698.37)	(8.84)	(18.32)	(5.05)
5		$NH_4S_2P\overline{OCMe_2CMe_2O}$	:1	$Cl_2Ru[S_2\overline{POCMe_2CMe_2O}]$	79	Above	371.6	8.21	16.50	18 31
	0.57	0.63		Brown powdery solid		400	(382.9)	(8.06)	(16.71)	(18.54)
6		$NH_4S_2P\overline{OCMe_2CMe_2O}$	1:2	$ClRu[S_2\overline{POCMe_2CMe_2O}]$	67.9	Above	547.86	10.85	22.83	6.11
	0.24	0.53		Purple powdery solid		400	(558.37)	(11.06)	(22.92)	(6.35)
7		$NH_4S_2P\overline{OCH_2CMe_2CH_2O}$	1:1	$Cl_2Ru[S_2\overline{POCH_2CMe_2CH_2O}]$	90.2	240	354.3	8.17	17.02	18.91
	0.83	0.86		Purple powdery solid			(368.9)	(8.37)	(17.34)	(19.24)
8		$NH_4S_2P\overline{OCH_2CMe_2CH_2O}$	1:2	$ClRu[S_2\overline{POCH_2CMe_2CH_2O}]_2$	89.0	260	512.4	11.54	24.03	6.44
	0.33	0.68		Vaint purple powdery solid			(530.6)	(11.64)	(24.12)	(6.69)

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Compound No.	Reactants (g)		Molar ratio	Product Physical state	Yield %	M.P.C	Mol. Wt.	Analysis % Found (Calc.)		
	$RuCl_3$	Amm. Dithiophosphate						P	S	Cl
9		$NH_4S_2P\overline{OCH_2CEt_2CH_2O}$	1:1	$Cl_2Ru[S_2P\overline{OCH_2CEt_2CH_2O}]$	84.1	345	383.7	7.63	15.84	17.65
	0.48	0.57		Brown powdery solid			(396.9)	(7.78)	(16.12)	(17.88)
10		$NH_4S_2P\overline{OCH_2CEt_2CH_2O}$	1:2	$ClRu[S_2P\overline{OCH_2CEt_2CH_2O}]_2$	83.9	320	564.1	10.44	21.71	5.787
	0.39	0.92		Purple powdery solid			(586.3)	(10.53)	(21.82)	(6 05)
11		$NH_4S_2P\overline{OCMe_2CH_2CHMeO}$	1:1	$Cl_2Ru[S_2P\overline{OCMe_2CH_2CHMeO}]$	78.8	390	366.2	7.87	16.63	18.36
	0.63	0.70		Brown powdery solid			(383.15)	(8.06)	(16.70)	(18.53)
12		$NH_4S_2P\overline{OCMe_2CH_2CHMeO}$	1:2	$ClRu[S_2P\overline{OCMe_2CH_2CHMeO}]_2$	83.4	–	543 2	1074	22.75	6.23
	0.38	0.85		Colourless volatile liquid			(558.7)	(11.06)	(22.90)	(6.35)
13		$Ru[S_2P(OPr^n)_2]_2$	1:2	$ClRu[S_2P(OPr^n)_2]_2$	87	–	380.2	8.25	16.42	18.36
	0.15	1.06		brown sticky solid			(384.9)	(8.02)	(16.62)	(18.44)
14		$Ru[S_2P(OPr^n)_2]_2$	2:1	$Cl_2Ru[S_2P(OPr^n)_2]_2$	89	–	552 8	10.73	22.53	6.49
	0.49	087		Dark brown sticky solid			(562.3)	(1094)	(22.76)	(6.69)
15		$Ru[S_2P\overline{OCH_2CMe_2CH_2O}]_3$	1:2	$ClRu[S_2P\overline{OCH_2CMe_2CH_2O}]_3$	92	260	515.4	11.43	23.87	6.49
	0.18	1.20		Vaint purple powdery solid			(530.6)	(11.54)	(24.12)	(6.69)

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TABLE II IR Spectral data for mixed chloro ruthenium (III) dialkyl and alkylenedithiophosphates

Comp. No.	*Compound*	ν *(P)-O-C*	ν *P-O-(C)*	*Ring vibration*	ν *P=S*	ν *P-S*	*Ru-S*	*Ru-Cl*
1	$Cl_2Ru[S_2P(OPr^n)_2]$	979s	857s	--	628m	536m	352m	311m
2	$ClRu[S_2P(OPr^n)_2]_2$	977s	847s	--	624m	541m	417m	350m
3	$Cl_2Ru[S_2P(OPh)_2]$	907s	846m	--	663m	566m	491w	305m
4	$ClRu[S_2P(OPh)_2]_2$	906s	769s	--	650s	571s	486m	303m
5	$Cl_2Ru[S_2\overline{POCMe_2CMe_2O}]$	1076m	841w	914m	630m	561w	486w	315s
6	$ClRu[S_2\overline{POCMe_2CMe_2O}]$	1003m	849m	953s	629s	581m	482w	301m
7	$Cl_2Ru[S_2\overline{POCH_2CMe_2CH_2O}]$	1046s	800m	985s	603m	538m	456m	397w
8	$ClRu[S_2\overline{POCH_2CMe_2CH_2O}]_2$	1037m	797w	983m	656m	536w	415m	395w
9	$Cl_2Ru[S_2\overline{POCH_2CEt_2CH_2O}]$	1066s	802m	982m	604m	542s	451w	370w
10	$ClRu[S_2\overline{POCH_2CEt_2CH_2O}]_2$	1064m	885m	966s	601m	546m	456m	374m
11	$Cl_2Ru[S_2\overline{POCMe_2CH_2CHMeO}]$	1033s	880m	949s	605s	511m	487w	309s
12	$ClRu[S_2\overline{POCMe_2CH_2CHMeO}]_2$	1035s	875m	953s	603s	514w	430m	319w

s=sharp, m=medium and w=weak

IR Spectra

The IR spectra of these mixed chloro dialkyldithiophosphate derivatives (Table II) have been measured in the range 4000- 200 cm^{-1} and assignments have been made on the basis of earlier reports.[16,17] The bands in the region 1076–977 and 857–797 cm^{-1} are assigned to ν (P)-O-C and νP-O-(C) stretching modes respectively. A strong band due to νP=S vibrations, observed in the region 670–662 cm^{-1} in the spectra of dialkyldithiophosphoric acids, is shifted to lower (~42 cm^{-1}) frequencies in these compounds. Bands of medium intensity present in the region 571–536 cm^{-1} have been assigned to νP-S asymmetric and symmetric stretching vibrations.

The bands present in the regions 1076–1003 and 880–797 cm^{-1} in the IR spectra of alkylenedithiophosphate derivatives may be assigned to ν (P)-O-C and νP-O-(C) stretching vibrations respectively. The bands in the region 985–914 cm^{-1} may be attributed to the ring vibrations of the dioxaphospholane and dioxaphosphorinane [18,19], which are probably coupled with C-C stretching vibrations. A sharp band present in the region 656–601 cm^{-1} is due to νP=S vibrations (showing a shift to lower frequency with respect to corresponding parent acid) and the bands in the region 581–511 cm^{-1} may be assigned to νP-S asymmetric and symmetric vibrations[20]. Bands of medium intensity present in the region 491–303 cm^{-1} are due to Ru-S[21] and Ru-Cl[22] stretching vibrations.

^{1}H NMR Spectra

The ^{1}H NMR spectra (Table III) of those derivatives recorded in $CDCl_3$, display the normal peaks due to alkoxy and glycoxy protons. The signals of hydrogen atoms, present at the -carbon atoms of P-O-C skeleton of alkyl or alkylene chains, become doubled due to coupling with ^{31}P nucleus.

^{31}P NMR spectra

Decoupled ^{31}P NMR spectra for representative products give a singlet for each compound in the range 82–61ppm. The single peak show also the purity of these complexes. It show deshielding to the extent of 0.4 – 5.95 ppm in case of I, II, V and VI. The deshielding in the complexes III, IV

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and VII are in the range 11.45 – 14.31 ppm with respect to the corresponding parent cyclic and a cyclic dithiophosphoric acids. This type of deshielding has been interpreted by Glidewell[23] to indicate the bidentate nature of dithiophosphato groups.

The above spectroscopic data discussed in this paper for the newly synthesized halide dialkyl and alkylenedithiophosphates of ruthenium, indicate the chelating nature of the dithiophosphate moieties. Considering the monomeric nature of all these derivatives, the monohalides bis(dialkyl and alkylenedithiophosphate) should contain penta-coordinated metal. The corresponding dihalide derivatives having a tetra-coordinated metal atom as shown in fig. 1and 2.

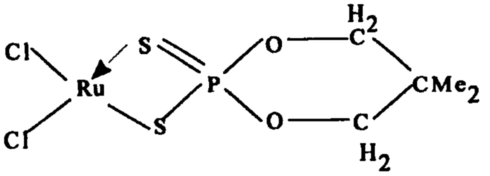

FIGURE 1 Suggested structure of tetracoordinated complexe

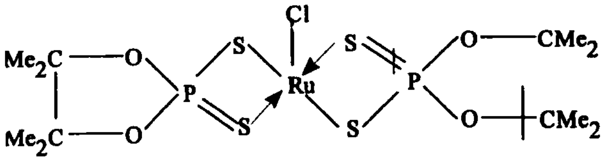

FIGURE 2 Suggested structure of Pentacoordinated complexe

EXPERIMENTAL

Precautions were taken to exclude moisture in some experimental manipulations. Solvents [benzene, chloroform and n-hexane] were dried by standard methods before use. Alcohols were distilled and dried. Glycols were distilled also. Dialkyl and alkylenedithiophosphoric acids and their ammonium salts were prepared by methods reported earlier[24,25]. Ruthenium trichloride (Strem) was used as received. Sulfur was estimated by Messen-

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ger's method as barium sulfate. Phosphorus was estimated as ammonium magnesium phosphate hexahydrated and chlorine was determined by the Volhard's method.

TABLE III ^1H NMR Spectral data for some mixed chloro ruthenium(III) dialkyl and alkylenedithiophosphates

Compound No.	*Compound*	*^1H Chemical shift (δppm) in $CDCl_3$*
1	$Cl_2Ru[S_2P(OPr^n)_2]$	0.95-.097, t, CH_3 (6H)
		1.5–2.04, m, CH_2 (4H)
		3.6–3.8, m, CH_2O (4H)
2	$ClRu[S_2P(OPr^n)_2]_2$	0.96-.097, t, CH_3 (12H)
		1.5–2.0, m, CH_2 (8H)
		3.6–3.8, m, CH_2O (8H)
3	$Cl_2Ru[S_2P(OPh)_2]$	7.26–7.29, m, C_6H_5 (10H)
4	$ClRu[S_2P(OPh)_2]_2$	6.42–7.25, m, C_6H_5 (20H)
5	$Cl_2Ru[S_2\overline{POCMe_2CMe_2O}]$	1.15–1.19, d, CH_3 (12H)
6	$ClRu[S_2\overline{POCH_2CMe_2CH_2O}]_2$	0.95–0.97, d, CH_3 (12H)
		3.6–3.9, d, CH_2O (8H)
7	$Cl_2Ru[S_2\overline{POCMe_2CH_2CHMeO}]$	1.4–1.5, m, $(CH_3)_2CH_2CH_3$(11H)
		4.8–5.2, m, CHO (1H)

Molecular weights were determined cryoscopically in benzene. IR spectra were recorded as Nujol mulls on a Perkin-Elmer 2000 FTIR in the range 4000 – 200 cm^{-1}, using CsI cells. ^1H NMR spectra were recorded in $CDCl_3$ on a Bruker AC 250 FT NMR using TMS as an internal standard. Phosphorous-31 spectra were also recorded using 85% H_3PO_4 as an external reference.

General methods of synthesis of various dialkyl and alkylenedithiophosphate derivatives of ruthenium(III)

a - Reaction between ruthenium trichloride and ammonium dialkyl and alkylenedithiophosphate in 1:1 and 1:2 molar ratio

Ruthenium trichloride and ammonium dialkyl and alkylenedithiophosphate in different stoichiometric ratios (1:1 and 1:2) were mixed, stirred, and refluxed for ~4 hours. Precipitated ammonium chloride was removed by filtration, followed by removal of the solvent. The desired product was washed several times by n-hexane and dried again under vacuum.

TABLE IV ^{31}P NMR Spectral data for some mixed chloro ruthenium (III) dialky and alkylenedithiophosphates

Compound No.	*Compound*	*^{31}P Chemical shift in δ ppm for parcent acid in $CDCl_3$ $CDCl_3$*	*^{31}P Chemical shift (δ ppm)*
1	$Cl_2Ru[S_2P(OPr^n)_2]$	82.40	82.03
2	$ClRu[S_2P(OPr^n)_2]_2$	--	81 85
3	$ClRu[S_2P(OPh)_2]_2$	80.13	67.61
4	$ClRu[S_2\overline{POCMe_2CMe_2O}]$	91.5	75.79
5	$ClRu[S_2\overline{POCH_2CMe_2CH_2O}]_2$	78.6	73.05
6	$Cl_2Ru[S_2\overline{POCH_2CEt_2CH_2O}]$	78.1	77.92
7	$Cl_2Ru[S_2\overline{POCMe_2CH_2CHMeO}]$	73.2	61.66

b - Reaction of ruthenium trichloride with the ammonium salt of dialkyl and alkylenedithiophosphoric acids in 1:3 molar ratio

On mixing ruthenium trichloride dissolved in dilute HCl with ammonium salt of dialkyl and alkylenedithiophosphoric acids dissolved in water. The mixture was stirred at room temperature (21°C) for ~6 hours and refluxed for half an hour with constant stirring. A 20-cm^3 volume of dichloromethane was added and transferred to a separation funnel. The mixture was shaken vigorously for 2 minutes and washed with distilled water. After the phase separation, the organic phase was dried under vacuum and washed with n-hexane and dried again under vacuum.

c - Disproportionation reaction of ruthenium chloride with ruthenium tris(di-n-propyl dithiophosphate) in 1:2 or 2:1 molar ratio

A mixture of ruthenium trichloride and ruthenium tris(di-n-propyl dithiophosphate) in 1:2 or 2:1 molar ratios was mixed in benzene and refluxed for ~4hours. The solvent was removed under reduced pressure giving the

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desired product, which washed several times with n-hexane and dried under vacuum.

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