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Novel β,δ-Triketonate Derivatives of Sn(II) and Sn(IV)

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Twelve new complexes of the type $Sn(II)(RCOCHCOCHCOR')$, $Sn(II)(RCOCH_2COCHCOR')_2$, $Cl_2Sn(IV)(RCOCHCOCHCOR')$ *and* $Sn(IV)(RCOCHCOCHCOR')_2$ ($R = R' = C_6H_5$ *or* CH_3; $R = C_6H_5$, $R' = CH_3$) *are synthesized and characterized by physical properties, elemental analyses, molecular weight determinations, conductance measurements and infrared and* ^{119m}Sn *Mössbauer data.*

Introduction

The β,δ-triketones and their Schiff bases are an important class of ligands, and homobimetallic complexes of β,δ-triketones [1–4] and heterobimetallic complexes of their Schiff bases [5–8] have recently been reported for transition metals, but no work appears to have been done with Sn(II) or Sn(IV).

In this paper we report the syntheses and characterization of novel Sn(II) and Sn(IV) complexes of the 1,5-disubstituted-1,3,5-pentanetriones.

Experimental

The reactions with Sn(II) and Sn(IV) were carried out under anhydrous conditions, in an atmosphere of oxygen-free nitrogen.

Benzene (BDH, AR) was kept over freshly drawn sodium wire for 24 hours and distilled. To the distillate a few drops of dried ethanol were added, and the mixture refluxed before the removal of the binary benzene–ethanol azeotrope. The fraction received at 80 °C was collected [9].

Anhydrous tin(II) chloride was prepared from tin(II) chloride dihydrate (BDH, AR). The latter was first kept under acetic anhydride, freshly distilled over P_2O_5, before use. The product was then washed successively with glacial acetic acid followed by dried diethylether [9]. Tin (IV) chloride (E. Merck) was used after distillation.

The three β,δ-triketones used here were prepared as follows:

Diacetylacetone (H_2DAA) was prepared by treating dehydroacetic acid with conc. HCl, followed by the addition of a hot barium hydroxide solution of pH 7. The resulting precipitate was dissolved in dil. HCl. The ligand was extracted from the latter solution with diethylether [10].

Dibenzoylacetone (H_2DBA) was prepared by the benzoylation of acetone with methylbenzoate in the presence of sodium hydride suspended in monoglyme [11].

Acetylbenzoylacetone (H_2ABA) was prepared by the benzoylation of acetylacetone with methylbenzoate in the presence of sodium hydride suspended in monoglyme [11].

Infrared spectra were recorded on a Perkin-Elmer 557 instrument in Nujol mulls. Tin-^{119m} Mössbauer spectra were recorded both at 77 K and room temperature on a Ranger Engineering, constant-acceleration spectrometer equipped with a proportional counter and with $Ca^{119m}SnO_3$ (New England Nuclear Corp.) used as the γ-ray source. The zero velocity was based upon $CaSnO_3$, and velocity calibrations on β-tin and natural iron foils. Standard, non-linear least-squares techniques were used to fit the data to Lorentzian curves.

Tin was estimated gravimetrically as tin(IV) oxide and chlorine was estimated volumetrically by Volhard's procedure.

Molecular weight determinations were made by both ebullioscopic and cryoscopic methods in boiling and cold benzene, respectively.

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TABLE I. Preparative and Physical Data for Mono- and Bis(β,δ-triketonato)Sn(II).

Reactants (g)			Molar ratio	Molecular Formula and State/Yield %	M.P. °C	Analyses %			Mol. Wt. Found (Calcd)
						Sn Found (Calcd)	C Found (Calcd)	H Found (Calcd)	
$SnCl_2$	H_2DBA[a]	NaOPr-i	1:1:2	$Sn(C_6H_5COCHCOCHCOC_6H_5)$	117d	30.98	53.30	4.05	335
1.42	2.00	1.25		light brown amorphous powder/85		(31.00)	(53.32)	(3.18)	(383)
$SnCl_2$	H_2DBA	NaOPr-i	1:2:2	$Sn(C_6H_5COCH_2COCHCOC_6H_5)_2$	121d	18.21	61.00	3.51	660
1.89	5.32	1.64		light brown amorphous powder/86		(18.28)	(62.88)	(3.72)	(649)
$SnCl_2$	H_2ABA[b]	NaOPr-i	1:1:2	$Sn(C_6H_5COCHCOCHCOCH_3)$	119d	36.90			298
1.85	1.99	1.60		light brown amorphous powder/83		(36.99)			(320)
$SnCl_2$	H_2ABA	NaOPr-i	1:2:2	$Sn(C_6H_5COCH_2COCHCOCH_3)_2$	112d	22.45			510
1.89	4.08	1.64		light brown amorphous powder/87		(22.60)			(525)
$SnCl_2$	H_2DAA[c]	NaOPr-i	1:1:2	$Sn(CH_3COCHCOCHCOCH_3)$	101d	45.78			241
1.89	1.42	1.64		brown amorphous powder/90		(45.86)			(258)
$SnCl_2$	H_2DAA	NaOPr-i	1:2:2	$Sn(CH_3COCH_2COCHCOCH_3)_2$	103d	29.01			355
1.89	2.82	1.64		light brown amorphous powder/85		(29.08)			(398)

[a] H_2DBA (Dibenzoylacetone) $C_6H_5COCH_2COC_6H_5$. [b] H_2ABA (Acetylbenzoylacetone) $C_6H_5COCH_2COCH_2COCH_3$.
[c] H_2DAA (Diacetylacetone) $CH_3COCH_2COCH_3$.

Synthesis of Tin(II) (1,5-Diphenyl-1,3,5-pentanetrionate)

Sodium isopropoxide (14 mmol) taken in dried benzene was added to the ligand (7 mmol). The resulting mixture was refluxed for 2 hrs. followed by the addition of tin(II) chloride (7 mmol) taken in tetrahydrofuran. The contents were refluxed for 2 hrs. and the sodium chloride formed was filtered and excess solvent removed *in vacuo*. The crude product on recrystallization from benzene afforded brown needles. The complex was found to be soluble in all common polar and nonpolar organic solvents.

Other tin(II) mono(β,δ-triketonates) and tin(II) bis(β,δ-triketonates) were prepared by the same procedure. The results of analyses and physical properties are given in Table I.

Synthesis of Dichlorotin(IV) (1,5-Diphenyl-1,3,5-pentanetrionate)

To a benzene solution of tin(IV) chloride (6 mmol) the ligand H_2DBA (6 mmol) was added in benzene dropwise to give an exothermic reaction with the evolution of HCl gas ceased (70 hrs.). After completion of the reaction, the solvent was stripped under vacuum to afford a shiny amorphous powder which is soluble in polar and non-polar organic solvents.

Other members of the series were also prepared by the same procedure. The results of analyses and physical data are given in Table II.

Synthesis of Bis(1,5-diphenyl-1,3,5-pentanetrionato)-Sn(IV)

Sodium-isopropoxide (16 mmol) taken in dried benzene was added to the ligand H_2DBA (8 mmol). The resulting mixture was refluxed for 2 hrs. followed by the addition to tin(IV) chloride (4 mmol) in benzene. The contents were refluxed for 2hrs., and the sodium chloride formed was filtered and excess of solvent removed under vacuum. The crude product on recrystallization from benzene afforded light yellow crystals.

Other compounds in this series were synthesized by the same procedure. The analytical results are given in Table II.

Results and Discussion

Tin(II)

The reaction of the di- and mono sodium (β,δ-triketonates) with tin(II) chloride can be represented as

$$Na_2^{+}(RCOCHCOCHCOR')^{2-} + SnCl_2 \rightarrow$$
$$\rightarrow \underset{(A)}{Sn(II)\,(RCOCHCOCHCOR')} + 2NaCl\downarrow \qquad (1)$$

$$2Na^{+}(RCOCH_2COCHCOR')^{-} + SnCl_2 \rightarrow$$
$$\rightarrow \underset{(B)}{Sn(II)\,(RCOCH_2COCHCOR')_2} + 2NaCl\downarrow \qquad (2)$$

The products of type A (1:1) complex are light brown colored amorphous powders, soluble and monomeric in freezing benzene. They melt at *ca.* 110–130 °C with decomposition and are non-conducting in dry nitrobenzene.

On comparing the infrared spectra (see Table IV) of the 1:1 complexes labeled A in Nujol mulls with those of the ligands it was observed that (i) a band at 1710–1680 cm^{-1} assigned to $\nu(C{=}O)$ shifts to lower energy at 1600 cm^{-1} in the solid complexes. This shows that all three carbonyl oxygens coordinate to the tin(II), and (ii) there is an increase in intensity of

TABLE II. Preparative and Physical Data for Dichloro-mono(β,δ-triketonato)Sn(IV) and Bis(β,δ-triketonato)Sn(IV).

Reactants (g)			Molar	Molecular Formula and State/Yield %	M.P.	Analyses % $Sn_{(Calcd)}^{Found}$	$Cl_{(Calcd)}$	$C_{(Calcd)}$	$H_{(Calcd)}$	Mol. Wt. Found (Calcd)
$SnCl_4$	H_2DBA[a]		1:1	$Cl_2Sn(C_6H_5COCHCOCHCOC_6H_5)$	97	26.11	15.61			425
1.75	1.80			brown amorphous powder/78		(26.15)	(15.64)			(453)
$SnCl_4$	H_2ABA[b]		1:1	$Cl_2Sn(C_6H_5COCHCOCHCOCH_3)$	115	30.21	18.07			406
2.60	2.04			grey amorphous powder/80		(30.29)	(18.11)			(391)
$SnCl_4$	H_2DAA[c]		1:1	$Cl_2Sn(CH_3COCHCOCHCOCH_3)$	105	36.02	21.00			322
2.60	1.42			brown amorphous powder/79		(36.10)	(21.28)			(328)
$SnCl_4$	H_2DBA	NaOPr-i	1:2:4	$Sn(C_6H_5COCHCOCHCOC_6H_5)_2$	95d	18.27		62.05	5.01	671
1.02	2.27	1.39		light brown amorphous powder/85		(18.34)		(63.08)	(3.73)	(647)
$SnCl_4$	H_2ABA	NaOPr-i	1:2:4	$Sn(C_6H_5COCHCOCHCOCH_3)$	100d	22.73				521
1.30	2.04	1.64		light brown amorphous powder/84		(22.75)				(523)
$SnCl_4$	H_2DAA	NaOPr-i	1:2:4	$Sn(CH_3COCHCOCHCOCH_3)_2$	91	31.00				401
1.30	1.42	1.64		brown amorphous powder/87		(31.16)				(381)

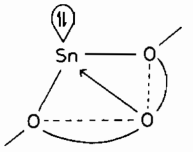
[a] H_2DBA (Dibenzoylacetone) $C_6H_5COCH_2COCH_2COC_6H_5$. [b] H_2ABA (Acetylbenzoylacetone) $C_6H_5COCH_2COCH_2COCH_3$. [c] H_2DAA (Diacetylacetone) $CH_3COCH_2COCH_2CH_3$.

TABLE III. Diagnostic Infrared Absorption Bands (cm^{-1}).[a]

Compound	νC=O	νC–O + C–C	νSn–O
H_2DBA	1680s	1595w	
Sn(II) (DBA)		1595w	
$Sn(II) (HDBA)_2$	1680s	1590m	600w
$Cl_2Sn(IV)$ (DBA)	1680m	1590m	570m
$Sn(IV) (DBA)_2$	1650m	1570m	640m
H_2ABA	1680s	1595w	
Sn(II) (ABA)		1600s	
$Sn(II) (HABA)_2$	1680s	1595m	595m
$Cl_2Sn(IV)$ (ABA)	1680m	1590m	570m
$Sn(IV) (ABA)_2$	1650–1630m	1560m	640w
H_2DAA	1680–1700s, b	1595w	
Sn(II) (DAA)		1595m	
$Sn(II) (HDAA)_2$	1680s	1590s	600w
$Cl_2Sn(IV)$ (DAA)	1680m	1590m	570m
$Sn(IV) (DAA)_2$	1650–1640s	1565m	640m

[a] s, sharp and strong; m, medium; w, weak; b, broad.

the ν(C=C) band at 1600 cm^{-1} in the ligand, on complexation because of the overlapping of ν(C=O) and ν(C=C) bands. This occurs in the other tin(II) derivatives as well. The coordination number of tin(II) is three [12, 13] in tin(II) sulfate [14] and potassium tris(formato)stannate(II) [15], both of which contain pyramidally tricoordinated tin(II). On this basis, and in order to produce three-coordination for tin in the present case, the β,δ-triketones must function as bis-enolic, tridentate ligands. The structure for these compounds is then likely to be similar to that of the tin(II)-carboxylates, $(RCOO)_2Sn(II)$ [16] in which the tin lone-pair occupying apical position of a tricoordinated, distorted pyramidal tin(II) *viz.*,



Tin-119m Mössbauer spectra of the (1:1) complexes, labeled (A), show isomer shifts (IS) in the tin(II) region [17] of 3.17–3.23 mms^{-1} and a quadrupole splitting (QS) in the range 1.97–1.99 mms^{-1} at 77 K. None of the compounds exhibited a Mössbauer effect at ambient temperature (see Table V) which would be suggestive of a polymeric lattice [18]. In each case the best fit to the spectrum was obtained by assuming a tin(II) doublet in the 1:1 complexes. The evidence favors identical structures for the series with a tricoordinated, distorted pyramidal tin environment, probably involving one bond longer than the other two [16], compatible with observed QS, arising from the distortion.

The (1:2) complexes, labeled B, are light brown colored amorphous powders, soluble and monomeric in benzene, and nonconducting in nitrobenzene.

TABLE IV. Infrared Bands (cm^{-1}) for the Ligands and Tin β,δ-Triketonate Derivatives.[a]

$C_6H_5COCH_2COCH_2COCH_3$	1710–1680 mb, 1600 wsp, 1585 wsp, 1540 w, 152 sw, 1495 w, 1450 msp, 1410–30 mb, 1330 msp, 1290 msp, 1180–90 mb, 1170 wsp, 1160 wsp, 1130 ssp, 1100 wsp, 1070 msp, 1030 ssp, 1000 wd, 930 sb, 810 ssp, 710 sb, 690 msp, 670 msp, 555 msp, 440 wsp.
$Sn(II)\ (C_6H_5COCHCOCHCOCH_3)$	1600 ssp, 1540 wsp, 1440 ssp, 1325 wsp, 1290 wsp, 1170 wb, 1125 wsp, 1100 wsp, 1070 wsp, 1025 wsp, 735 ssp, 800 msp, 710 ssp, 685 wsp, 670 wsp, 550 msp.
$Sn(II)\ (C_6H_5COCH_2COCHCOCH_3)_2$	1680 ssp, 1595 msp, 1375 msp, 1325 wb, 1295 msp, 1170 wb, 1125 wb, 1100 wsp, 1070 wb, 1020 wsp, 735 sb, 800 msp, 710 ssp, 685 wsp, 670 wsp, 595 wb, 555 mb.
$Cl_2Sn(IV)\ (C_6H_5COCHCOCHCOCH_3)_2$	1680 msp, 1590 ssp, 1530–1520 mb, 1400–1380 mb, 1180 msp, 1070 wsp, 1025 msp, 935 msp, 825 wsp, 810 wb, 715 ssp, 685 wb, 750 msp.
$Sn(IV)\ (C_6H_5COCHCOCHCOCH_3)_2$	1650–1630 mb, 1560 msp, 1530 msp, 1400–1380 wb, 1180 msp, 1070 wsp, 1025 msp, 935 mb, 825 wb, 810 wb, 715 ssp, 685 wb, 640 wb.
$C_6H_5COCH_2COCH_2COC_6H_5$	3060–2820 wb, 1710–1680 sb, 1600 msp, 1580 msp, 1555 wsp, 1540 wsp, 1490 w, 1450 ssp, 1420 ssp, 1320 ssp, 1290 ssp, 1210 wsp, 1180 msp, 1130 ssp, 1100 wsp, 1070 ssp, 1030 ssp, 1000 wsp, 930 sb, 805 s and 815 d, 710 ssp, 690 msp, 670 ssp, 555 ssp, 470 msp.
$Sn(II)\ (C_6H_5DODHCOCHCOC_6H_5)$	1595 ssp, 1450 ssp, 1375 ssp, 1320 wd, 1290 msp, 1170 wb, 1125 wsp, 1100 wsp, 1070 wsp, 1025 wsp, 730 sb, 800 ssp, 710 ssp, 685 wsp, 670 wsp, 555 mb.
$Sn(II)\ (C_6H_5COCH_2COCHCOC_6H_5)_2$	1680 msp, 1590 msp, 1375 msp, 1325 wsp, 1290 msp, 1170 wb, 1125 wb, 1100 wsp, 1070 wsp, 1020 wsp, 730 sb, 800 msp, 710 ssp, 685 wsp, 670 wsp, 600 wb, 555 mb.
$Cl_2Sn(IV)\ (C_6H_5COCHCOCHCOC_6H_5)$	1680 msp, 1590 msp, 1520 mb, 1400 sb, 1320 wsp, 1280 wb, 1180 msp, 1100 wsp, 1070 wsp, 1025 wsp, 1000 wsp, 930 msp, 805 msp, 710 ssp, 680 wsp, 620–610 wb, 570 mb, 490 sb.
$Sn(IV)\ (C_6H_5COCHCOCHCOC_6H_5)_2$	1650 mb, 1570 msp, 1525 msp, 1400 ssp, 1320 wsp, 1280 wb, 1185 mb, 1100 wsp, 1070 wsp, 1025 wsp, 1000 wsp, 930 msp, 710 ssp, 640 wb.
$CH_3COCH_2COCH_2COCH_3$	1710 ssp, 1650 w, 1600 msp, 1420 msp, 1390 msp, 1240 msp, 1190 msp, 1160 msp, 1040 msp, 895 mb, 530 wsp, 510 wsp.
$Sn(II)\ (CH_3COCHCOCHCOCH_3)$	1690 sh, 1505 ssp, 1400 msp, 1305 mb, 1255 wsp, 1220 wb, 1165 mb, 1095 mb, 1015 msp.
$Sn(II)\ (CH_3COCH_2COCHCOCH_3)_2$	1700 msp, 1590 ssp, 1400 msp, 1310 mb, 1255 wb, 1225 wb, 1170 mb, 1095 msp, 1020 msp, 600 wb.
$Cl_2Sn(IV)\ (CH_3COCHCOCHCOCH_3)$	1710–1680 mb, 1590 msp, 1395 ssp, 1310 mb, 1250 wsp, 1225 wb, 1710 msp, 1095 wsp, 1020 msp, 570 mb.
$Sn(IV)\ (CH_3COCHCOCHCOCH_3)_2$	1650–1640 ssp, 1565 msp, 1390 ssp, 1310 mb, 1255 wsp, 1220 wb, 1165 mb, 1095 mb, 1015 mb, 640 mb.

[a] s = strong, m = medium, w = weak, sp = sharp, b = broad, sh = shoulder, d = doublet.

Infrared spectra of the complexes of the type B (see Table IV) show the following changes: (i) the band ν(C=O) at 1710–1680 cm^{-1} in the ligand is split into two in the complex, retaining one band at 1680 cm^{-1}, showing the presence of one free carbonyl group, while the other merges into the ν(C=O) band at 1600 cm^{-1}, increasing its intensity. Thus the ligand functions in a mono-enolic bidentate fashion in the 1:2 complexes, labeled B, in which the tin is surrounded by four oxygens, two of them ester-bonded and two datively bonded to tin. In view of this, a four-coordinated, square-pyramidal geometry is suggested for the compounds of the type B, *viz.*,

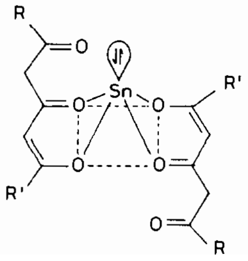


TABLE V. Tin-119 Mössbauer Data for Complexes of $C_6H_5COCHCOCHCOR$ (R = CH_3, C_6H_5) at 77 K in mm s^{-1}.

Sample	IS ± 0.03	QS ± 0.06	Γ−	Γ+
Sn(II) ($C_6H_5COCHCOCHCOC_6H_5$)	3.17 0.04[a]	1.99 0.56	1.15 1.11	1.13 1.11[b]
Sn(II) ($C_6H_5COCHCOCHCOCH_3$)	3.23 0.08[a]	1.97 0.63	1.22 1.23	1.19 1.23[b]
Cl_2Sn(IV) ($C_6H_5COCHCOCHCOCH_3$)	0.17	0.57	1.10	1.10[b]
Sn(IV) $(C_6H_5COCHCOCHCOCH_3)_2$	0.03 0.07[c]	0.65	1.07 1.35[c]	1.07[b]

[a] An oxidation product, presumably the Sn(IV) bis-ligand complex, produced on exposure to air. [b] Fit constrained to give equal linewidths. [c] Room temperature data.

Tin(IV)

Reactions of the β,δ-triketones with tin(IV) chloride are represented as

$$SnCl_4 + RCOCH_2COCH_2COR' \xrightarrow[70\ hrs]{\Delta}$$

$$Cl_2Sn(IV)\,(RCOCHCOCHCOR') + 2HCl\uparrow \quad (3)$$
$$(C)$$

$$SnCl_4 + 2Na_2(RCOCHCOCHCOR') \xrightarrow[2\ hrs]{\Delta}$$

$$Sn(IV)\,(RCOCHCOCHCOR')_2 + 4NaCl\downarrow \quad (4)$$
$$(D)$$

The (1:1) complexes of Sn(IV), labeled C, are brown-colored, shiny hygroscopic, amorphous powders, soluble, and monomeric in freezing benzene and non-conducting in dry nitrobenzene.

Infrared spectra of the (1:1) complexes of Sn(IV), (C), show characteristic bands at 1680, 1590 and 570 cm^{-1}, corresponding to free ν(C=O), bonded ν(C=O) + ν(C=C) (overlapping) and ν(Sn–O) bands, respectively, in the complexes. The observation of the band at 1680 cm^{-1} shows that either one free or very weakly bonded carbonyl group is present in the complexes. We interpret the increase in the intensity of the band at 1590 cm^{-1} on complexation as owing to the overlap of a bonded carbonyl group band with the one originating from (C=C) group. A new band appears in the spectra of all the complexes at 570 cm^{-1}, assigned to ν(Sn–O). On the basis of above observations, it can be concluded that β,δ-triketone may coordinated either as a bidentate or as tridentate bis-enolic ligand.

The Mössbauer spectrum of Cl_2Sn(IV) ($CH_3COCHCOCHCOC_6H_5$) gives no resonance at room temperature, but at 77 K gives a narrow doublet pattern in the tin(IV) region corresponding to a tin atom highly coordinated by electronegative atoms. The ratio (ρ) of the QS to the IS [18] is 3.3 in this case, which together with the low IS forces the conclusion that the tin(IV) alone is higher than four-coordinated. Three isomers are possible in a trigonal bipyramidal $SnCl_2O_3$ geometry containing two, one or no axial-chlorine atoms, but no conclusions can be drawn from these data concerning which is present. It is assumed that the ligand can span the axial-positions. Values of the IS and QS are given in Table V.

The (1:2) complexes, labeled (D), are brown amorphous powders, soluble and monomeric in benzene and non-conducting in dry nitrobenzene.

The infrared spectra of these complexes show characteristic bands in the regions 1650–1630, 1570–1560 and 640 cm^{-1}, assigned to the bonded carbonyl group, ν(C=C) + ν(C=O) groups, and the tin-oxygen bond. The absence of absorption at 1680 cm^{-1} arising from free carbonyl groups in the spectra of the 1:2 complexes indicates that all six oxygen atoms are bonded to tin(IV), and a six-coordinated, octahedral structure is proposed. There are two possible forms, the facial- or meridional-isomer.

The Mössbauer spectrum of Sn(IV) $(CH_3COCHCOCHCOC_6H_5)_2$ was studied both at room temperature and 77 K and the data listed in Table IV. At 77 K this derivative gave a narrow doublet pattern in the tin(IV) region corresponding to a tin atom highly coordinated by electronegative atoms and the resonance was observable at room temperature. Although ^{119m}Sn Mössbauer spectra of monomeric tin complexes can generally only be recorded at 77 K, yet six-coordination is sometimes accompanied by a measurable resonance at room temperature [18]. The QS observed for this derivative rules out the facial-isomer which would generate no electric field gradient at the tin nucleus [19].

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