Tellurium Complexes with Substituted Chalcones

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Several complexes of tellurium(IV) with substituted benzalacetophenone (chalcones) were synthesized and characterized by elemental analyses, conductivity measurements, and infrared (IR) spectroscopic data. Elemental analyses confirmed a 1:2 (metal:ligand) stoichiometry. The complexes were shown to be biologically active as evidenced by pharmacological tests.

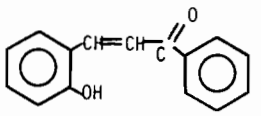
Introduction

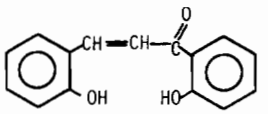
In recent years several papers have dealt with the preparation of transition metal complexes of benzalacetophenones (*chalcones*) [1–4]. However, no systematic research has been reported on the syntheses of complexes of substituted chalcones with tellurium(IV). A study was undertaken in order to synthesize, characterize and determine the biological activity of a number of tellurium(IV) complexes of chalcones. The complexes were characterized using infrared and conductivity measurements. The biological activity was determined using bacteriostatic tests.

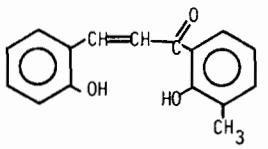
Experimental

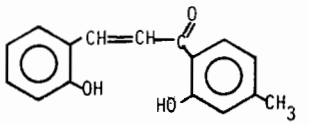
All the chemicals used were of reagent grade. The chalcones were prepared by reacting salicylaldehyde with substituted acetophenone according to a method reported in the literature [5].

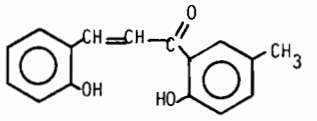
The following chalcones were prepared:

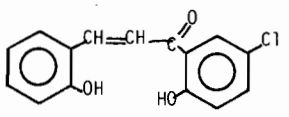
I (A)  2-hydroxybenzalacetophenone

II (B)  2,2′-dihydroxybenzalacetophenone

III (C)  2,2′-dihydroxy-3-methylbenzalacetophenone

IV (D)  2,2′-dihydroxy-4-methylbenzalacetophenone

V (E)  2,2′-dihydroxy-5-methylbenzalacetophenone

VI (F)  2,2′-dihydroxy-5-chlorobenzalacetophenone

The tellurium(IV) complexes were prepared by mixing tellurium tetrachloride with the ligands in dry benzene in the molar ratio of 1:2, with vigorous shaking. The resulting complex was then filtered, washed repeatedly with anhydrous benzene using a Soxhlet extractor, and finally dried under vacuum

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TABLE I. Analytical and Physical Data for Chalcone Complexes of Tellurium.

Ligand	Complex No.	Empirical formula of the complex	M.P. °C	%Te[a]	%Cl[a]	Molecular weight	Molar Cond. ohm^{-1} cm^2 $mole^{-1}$
A	I	$(C_{15}H_{11}O_2)_2TeCl_4$	141	17.85 (17.84)	19.80 (19.85)	720.40 (715.44)	124.60
B	II	$(C_{15}H_{11}O_3)_2TeCl_4$	145	18.92 (18.86)	10.56 (10.50)	670.00 (676.44)	68.75
C	III	$(C_{16}H_{14}O_3)_2TeCl_4$	151	16.49 (16.41)	18.30 (18.27)	700.00 (777.44)	120.80
D	IV	$(C_{16}H_{14}O_3)_2TeCl_4$	140	16.50 (16.41)	18.33 (18.27)	698.30 (777.44)	125.20
E	V	$(C_{16}H_{14}O_3)_2TeCl_4$	104	16.53 (16.41)	18.28 (18.27)	800.00 (777.44)	130.30
F	VI	$(C_{15}H_{11}O_3Cl)_2TeCl_4$	150	15.99 (16.06)	17.85 (17.87)	702.00 (794.44)	61.70

[a]Figures in the parentheses are theoretical yields.

over P_2O_5. Table I correlates each ligand (A–F) with its corresponding tellurium complex (I–VI).

Elemental analyses were carried out by a procedure discussed elsewhere [6]. Tellurium was estimated as the tellurium metal, chlorine as the silver chloride precipitate, while barium was estimated as the barium sulfate salt. The method of Kjeldahl was used to determine the nitrogen content of the complexes.

The molecular weights were obtained by using an ebullioscopic technique utilizing nitrobenzene as a solvent [7].

Conductivities were measured in dimethyl formamide (DMF) using an Elico-CM-82 conductivity bridge with a cell having a cell constant of 0.829 cm^{-1}. All conductivity measurements were performed at room temperature using 10^{-3} *M* solutions of the complex.

The infrared spectra (IR) from 4000 to 200 cm^{-1} were obtained using a Perkin-Elmer 180 spectrophotometer. Samples were prepared as KBr pellets.

The antibacterial activity test was adapted from a method used by Chaturvedi and coworkers [8]. The test compounds were dissolved in dimethyl formamide, then added to a nutrient agar for bacteria giving a final concentration of 200 μg/ml. The extent of inhibition was measured in millimeters using the zone of inhibition produced after 24 hours. The bacteriostatic properties of the substituted chalcones of tellurium were compared with those of uncoordinated ligands.

Results and Discussion

Analytical Data

All the complexes are colored and insoluble in common organic solvents, but soluble in methanol, DMF, and DMSO. The elemental analyses (see Table I) agree well with 1:2 stoichiometry (metal:ligand). The ebullioscopic molecular weight determination indicates that all the complexes are monomeric. The molar conductivities for the majority of the complexes are in the range of 120–131 ohm^{-1} cm^2 $mole^{-1}$. These values are lower than those expected for 1:2 electrolytes. Two complexes have molar conductivities in the range, 62–69 ohm^{-1} cm^2 $mole^{-1}$ suggesting 1:1 electrolyte behavior [9].

Infrared Spectra

Important infrared frequencies and their assignments are tabulated and available upon request from the author to whom all correspondence should be addressed.

Three characteristic bands are observed in the region, 3300–2600 cm^{-1} for the ligands B–F. The medium intensity band at 3300–3200 cm^{-1} is assigned to the intermolecular ν(O–H) of the benzal moiety; the band at 2950–2880 cm^{-1} is due to ν(C–H); and the broad weak band at 2675–2650 cm^{-1} is attributed to an intramolecular ν(O–H) originating with the acetophenone moiety [10]. Ligand A does not have a band at 2650 ± 25 cm^{-1} since there is no 2-hydroxy group associated with the acetophenone moiety.

In all the tellurium complexes (I–VI), the band associated with ν(O–H) of the benzal moiety does not disappear in the IR spectra indicating non-participation of the 2-hydroxy group in the bonding. In tellurium complex II, the band at 2650 cm^{-1} in the ligand disappears, indicating that the hydroxyl group of the acetophenone moiety is involved in the bonding to tellurium. The remaining tellurium complexes III–VI do not show any change in the band at 2650 ± 25 cm^{-1}.

TABLE II. Antibacterial Activity of Chalcones and Their Tellurium(IV) Complexes.

Ligand/Complex	Zone of inhibition in nm		
	Organisms		
	E. coli	*B. subtilis*	*S. aureus*
2-Hydroxybenzolacetophenone	24	22	36
Tetrachlorobis[2-hydroxybenzolacetophenone]tellurium(IV)	45	30	33
2,2'-Dihydroxybenzalacetophenone	16	17	20
Dichlorobis[2,2'-dihydroxybenzalacetophenone]tellurium(IV)	22	20	38
2,2'-Dihydroxy-5-methylbenzalacetophenone	18	22	38
Tetrachlorobis[2,2'-dihydroxy-5-methylbenzalacetophenone]tellurium(IV)	22	22	38
2,2'-Dihydroxy-5-chlorobenzalacetophenone	18	22	30
Tetrachlorobis[2,2'-dihydroxy-5-chloro-benzalacetophenone]tellurium(IV)	34	22	34

In the ligands, an intense band around 1650–1640 cm^{-1} is assigned to ν(C=O) [11]. The same band in the tellurium complexes is shifted to 1640–1625 cm^{-1}, suggesting that the carbonyl oxygen is coordinated to the tellurium.

Intense bands around 1615–1460 cm^{-1} are assigned to aromatic and ethylenic C=C vibrations [5]. Bands in the region 1280–1265 cm^{-1} are assigned to a phenolic ν(C–O) [12]. Complex II exhibits a high frequency shift of 15 cm^{-1} relative to ligand B. This further substantiates the involvement of the acetophenone hydroxy group in the bonding.

In the tellurium complexes, bands in the range 590–520 cm^{-1}, are assigned to ν(Te–O) [13]. The intense band at 320 cm^{-1} is assigned to ν(Te–Cl) [14, 15],

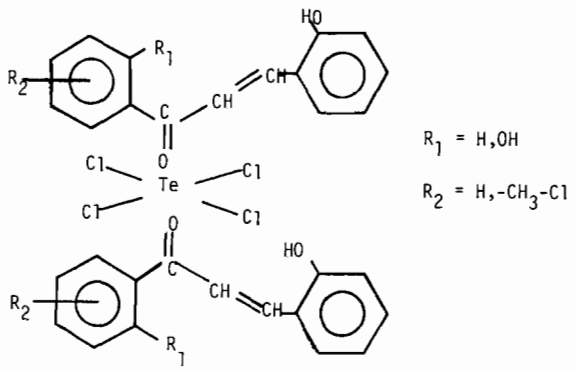
Antibacterial Activity Test

The antibacterial activity of the substituted chalcones and their complexes are listed in Table II. The compounds were screened for their antibacterial activity using three micro-organisms; namely, *E. Coli* (gram negative); *B. Subtilis* and *S. Aureus* (gram positive). These bacterial strains were chosen since they are known commensals and pathogens of human beings. The results show that the complexes of tellurium are more active in the inhibition of *E. Coli* than the corresponding chalcone. The tellurium complexes are more active than the parent chalcones in some cases when assessing the antibacterial activity against *B. Subtilis* and *S. Aureus*. The enhanced activity of the tellurium complexes may be due to their enhanced lipid solubility and subsequent cellular penetration.

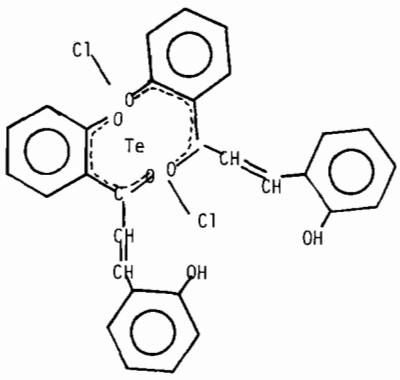
Conclusions

A few complexes of Tellurium(IV) with substituted benzalacetophenones have been synthesized and characterized. The elemental analyses indicate that these complexes have a 1:2 stoichiometry. Based on the IR data, and the foregoing results, the following tentative structures have been proposed:

Complexes I, III - VI



Complex II



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