

Some (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te and (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te Molecular Adducts with Monodentate Ligands

SHEKHAR SRIVASTAVA*, HARI S. GUPTA and DEEPA GAUTAM

*School of Studies in Chemistry
Jiwaji University, Gwalior-474 011, India*

Twenty molecular adducts of (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te and (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te with various monodentate ligands of the type RR'Te-L have been synthesised and analysed to assign their structure by elemental analysis, molar conductivity, IR and X-ray photoelectron spectroscopy.

INTRODUCTION

In our previous communication¹, we have reported the reaction of (C₆H₅)₂Te and (*p*-MeOC₆H₄)₂Te with various monodentate ligands. This paper deals with further extension of previous communication¹, for studying the reaction of (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te and (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te with various monodentate ligands *i.e.*, pyridine, pyridine-N-oxide, morpholine, 4-picoline, trimethylamine, dimethylsulphoxide, dimethylformamide, triphenylphosphine sulfide, triphenyl phosphine oxide and triphenylphosphine and their structural determination by various physico-chemical techniques.

EXPERIMENTAL

The (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te and (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te were prepared, purified and characterised according to literature procedure².

1 mmol of (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te or (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te in 25 mL of dry chloroform is refluxed with 1 mmol of either pyridine, pyridine-N-oxide, morpholine, 4-picoline, trimethylamine, dimethyl sulphoxide, dimethyl formamide, triphenylphosphine sulfide, triphenylphosphine oxide or triphenylphosphine for 3 h. The reacted mixture was rotary evaporated and the concentrated solution was allowed to stay overnight in the deep freeze. The crystals obtained were washed with petroleum ether (60–80°C) and dried in vacuum.

Conductance measurements were obtained in acetone at room temperature using a Digisun Electronic Conductivity Bridge. Infrared spectra were recorded in CsI on a Perkin-Elmer 457 spectrometer. The X-ray photoelectron spectra were recorded on a VG Scientific ESCA-3MKII electron spectrometer. The MgK α X-ray line (1253.6 eV) was used for photo-excitation. The Cu2p_{3/2} (BE = 932.8 \pm 0.2) and Au4f_{7/2} (BE = 83.8 \pm 0.1) lines were used to calibrate the

Present address: Department of Chemistry, University of Allahabad, Allahabad (U.P.).

instrument and $\text{Ag}3d_{3/2}$ ($\text{BE} = 368.2$) was used for cross-checking³. All the spectra were recorded using the same spectrometer parameters of 50 eV pass energy and 4 mm slit width. The reduced full width at half maximum (FWHM) at the $\text{Au}4f_{7/2}$ ($\text{Be} = 83.8$ eV) level under these conditions was 1.2 eV.

RESULTS AND DISCUSSION

All the molecular adducts were air-stable. Elemental analyses were within $\pm 0.5\%$ for C, H and N. The observed molar conductances of all the molecular adducts in acetone were in the range of 20–30 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$, indicating that all these prepared complexes are non-ionic species⁴ (Table-1).

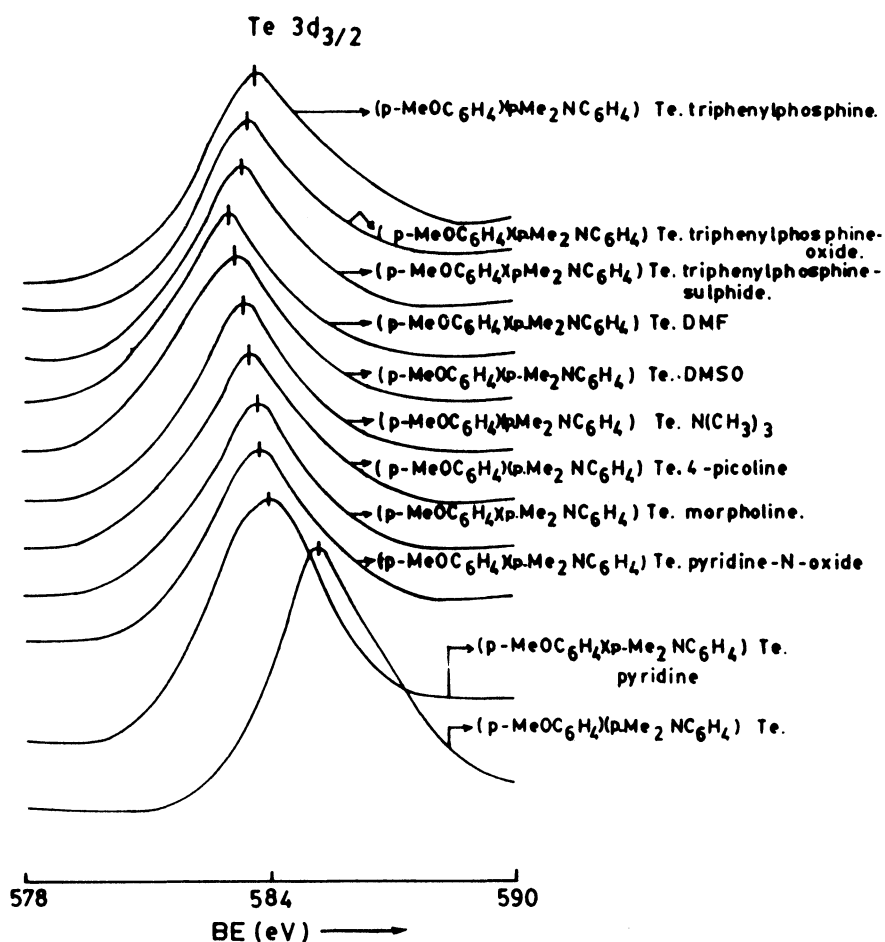


Fig. 1 Te 3d_{3/2} Photoelectron peak in $(p\text{-MeOC}_6\text{H}_4)_2(p\text{-Me}_2\text{NC}_6\text{H}_4)\text{Te}$ and $(p\text{-MeOC}_6\text{H}_4)(p\text{-Me}_2\text{NC}_6\text{H}_4)\text{Te}\cdot\text{L}$ complexes

TABLE-1. ANALYTICAL DATA OF (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te AND (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te MOLECULAR ADDUCTS

S. No.	Complex	% Found (Calcd.)				λ_m^* ohm ⁻¹ cm ² mole ⁻¹
		Te	C	H	N	
1.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te ·pyridine	29.3 (29.42)	55.2 (55.35)	5.0 (5.07)	6.4 (6.45)	22
2.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te ·pyridine-N-oxide	28.1 (28.38)	53.2 (53.38)	4.4 (4.89)	6.2 (6.22)	24
3.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te ·morpholine	28.4 (28.89)	51.4 (51.63)	5.4 (15.88)	6.2 (6.34)	27
4.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te ·4-picoline	28.4 (28.50)	56.1 (56.30)	5.1 (5.36)	6.0 (6.25)	26
5.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te ·N(CH ₃) ₃	30.4 (30.85)	52.1 (52.22)	6.1 (6.28)	6.4 (6.76)	28
6.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·DMSO	29.4 (29.49)	47.0 (47.15)	5.2 (5.31)	3.0 (3.23)	30
7.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·DMF	29.4 (29.84)	50.4 (50.51)	5.3 (5.61)	6.4 (6.54)	29
8.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·Ph ₃ PS	19.4 (19.67)	61.0 (61.05)	4.4 (4.93)	2.0 (2.15)	21
9.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·Ph ₃ PO	20.0 (20.17)	62.4 (62.59)	5.0 (5.05)	2.1 (2.21)	20
10.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·Ph ₃ P	20.2 (20.69)	64.5 (64.22)	5.5 (5.18)	2.2 (2.27)	30
11.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·pyridine	28.4 (28.50)	56.2 (56.36)	5.2 (5.36)	6.0 (6.25)	21
12.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·pyridine- N-oxide	27.4 (27.52)	54.2 (54.35)	5.0 (5.17)	6.0 (6.03)	30
13.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te· morpholine	28.0 (28.00)	52.3 (52.67)	6.0 (6.14)	6.0 (6.14)	29
14.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·4picoline	27.3 (27.64)	57.0 (57.19)	5.4 (5.63)	6.0 (6.06)	25
15.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·N(CH ₃) ₃	29.4 (29.84)	53.2 (53.22)	6.4 (6.54)	6.4 (6.54)	27
16.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·DMSO	28.4 (28.57)	48.2 (48.36)	5.4 (5.59)	3.0 (3.13)	22
17.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·DMF	28.4 (28.89)	51.4 (51.63)	5.4 (5.88)	6.2 (6.39)	20
18.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·Ph ₃ PS	19.0 (19.25)	61.4 (61.57)	5.0 (5.13)	2.0 (2.11)	25
19.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·(C ₆ H ₅) ₃ PO	19.4 (19.73)	63.0 (63.09)	5.0 (5.25)	2.0 (2.16)	28
20.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te·Ph ₃ P	20.0 (20.23)	64.4 (64.70)	5.2 (5.39)	2.0 (2.22)	30

*Solvent—Acetone.

$\nu(\text{C}=\text{N})$ modes in 4-picoline absorbs at 1569 cm⁻¹, shifted to 1640 cm⁻¹ on complexation⁵. $\nu(\text{S}=\text{O})$ in DMSO absorbs at 1045 cm⁻¹ shifted to 948 cm⁻¹ in

these RR'Te-DMSO. This lowering is due to the coordination of oxygen atom of DMSO to tellurium metal⁶. $\nu(\text{P}=\text{O})$ vibration of free triphenylphosphine oxide around 1192 cm^{-1} moves to a lower frequency in molecular adducts, showing coordination of triphenylphosphine oxide to the tellurium metal ion through oxygen atom^{7, 8}. $\nu(\text{C}=\text{O})$ vibration of free DMF at $1665 \pm 15\text{ cm}^{-1}$ moves to $1635 \pm 15\text{ cm}^{-1}$ upon complexation indicating coordination *via* oxygen atom in RR'Te-DMF.⁹ In far IR, all the molecular adducts have shown bands at $260\text{--}220\text{ cm}^{-1}$, which may be assigned as $\nu(\text{Te}-\text{C})$ ¹⁰.

The binding energies (eV) of (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te and (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te and their molecular adducts for Te3d_{3/2, 5/2} photoelectron peaks are listed in Table-2. It was observed that the binding energies of Te3d_{3/2, 5/2} in the (*p*-MeOC₆H₄)(*p*-Me₂NC₆H₄)Te and (*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te were higher than prepared by their molecular adducts. One can conclude from these XPS data that the electron density on tellurium metal ion has been increased due to coordination of ligands with tellurium metal ion³ (Table-2 and Fig. 1–2).

TABLE-2
BINDING ENERGIES (eV) OF (*p*-MeOC₆H₄)(Me₂NC₆H₄)Te AND
(*p*-EtOC₆H₄)(*p*-Me₂NC₆H₄)Te AND THEIR MOLECULAR ADDUCTS

S. No.	Complex	Te	
		Te3d _{3/2}	Te3d _{5/2}
1.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te	585.2	575.0
2.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-pyridine	584.0	574.2
3.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-pyridine-N-oxide	583.8	574.0
4.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-morpholine	583.8	573.8
5.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-4-picoline	583.6	573.6
6.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-N(CH ₃) ₃	583.4	573.8
7.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-DMSO	583.2	573.8
8.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-DMSO	583.0	573.6
9.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-Triphenylphosphine sulfide	583.4	573.6
10.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-Triphenylphosphine oxide	583.6	573.6
11.	(<i>p</i> -MeOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-Triphenylphosphine	583.8	573.6
12.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te	585.4	575.8
13.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-pyridine	584.0	574.0
14.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-pyridine-N-oxide	583.8	574.0
15.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-morpholine	583.6	573.8
16.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-4 picoline	583.6	573.8
17.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-N(CH ₃) ₃	583.8	573.6
18.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-DMSO	583.8	573.6
19.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-DMF	583.8	573.6
20.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-Triphenylphosphine sulfide	583.6	573.8
21.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-Triphenylphosphine oxide	583.6	573.8
22.	(<i>p</i> -EtOC ₆ H ₄)(Me ₂ NC ₆ H ₄)Te-Triphenylphosphine	583.6	573.8

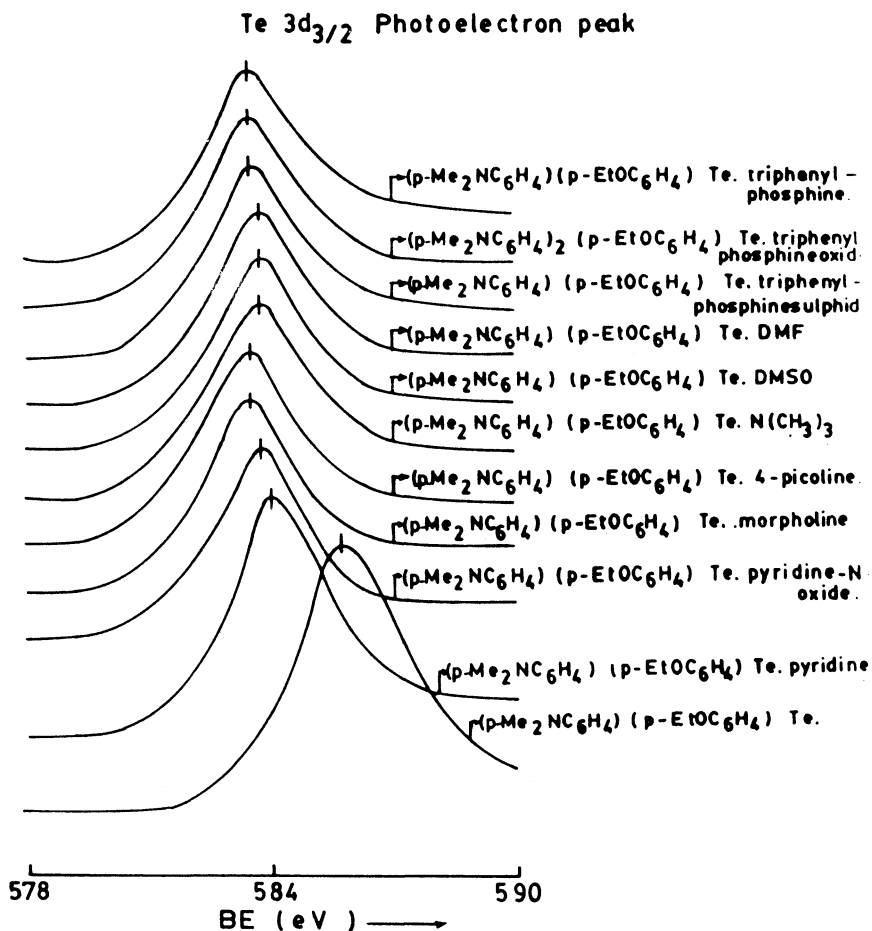


Fig. 2 Te 3d_{3/2} Photoelectron peak in $(p\text{-Me}_2\text{NC}_6\text{H}_4)(p\text{-EtOC}_6\text{H}_4)\text{Te}$ and $(p\text{-Me}_2\text{NC}_6\text{H}_4)(p\text{-EtOC}_6\text{H}_4)\text{Te}\cdot\text{L}$ complexes

On the basis of above physico-chemical techniques results, it is possible to assign a structure for each adduct as shown in Fig. 3 (a and b) and trigonal bipyramidal geometry as shown in Fig. 4.

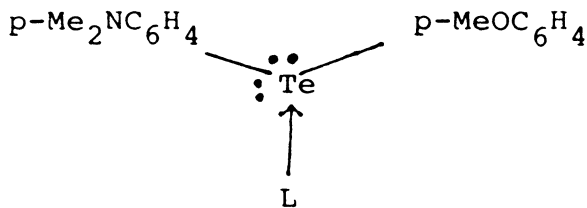


Fig. 3 (a)

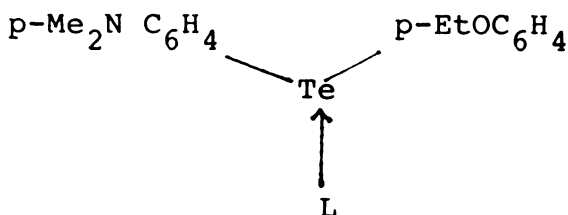


Fig. 3 (b)

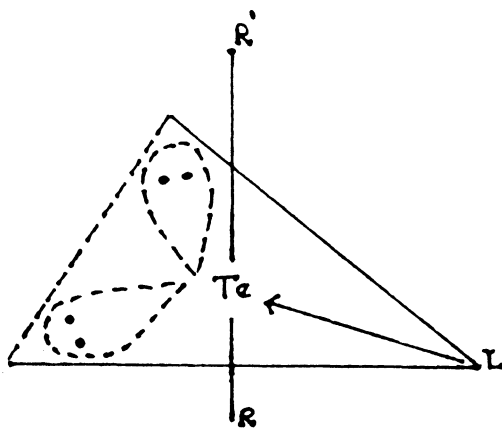


Fig. 4

R = (*p*-MeOC₆H₄), R' = (*p*-Me₂NC₆H₄) or
 R = (*p*-EtOC₆H₄), R' = (*p*-Me₂NC₆H₄)

ACKNOWLEDGEMENT

Sincere thanks of the authors are due to UGC, New Delhi for financial assistance to one of them (H.S.G.).

REFERENCES

1. Shekhar Srivastava, Hari Shankar Gupta nad Deepa Gautam, *J. Indian. Chem. Soc.* (communicated).
2. N. Petraghani, *Tetrahedron*, **12**, 219 (1961).
3. Shekhar Srivastava, *Applied Spectros. Rev.*, **22**, 401 (1986).
4. W.L. Greary, *Coord. Chem. Rev.*, **13**, 47 (1971).
5. D. Cook, *Can. J. Chem.*, **12**, 2523 (1964).
6. J. Gopalakrishnan and C.C. Patel, *J. Sci. Ind. Res.*, **27**, 475 (1968).
7. N.M. Karayannis, C.M. Mikulski, M.J. Strucko, L.L. Pyttewski and M.M. Labes, *J. Inorg. Nucl. Chem.*, **32**, 2629 (1970).
8. D.M.L. Goodgame and F.A. Cotton, *J. Am. Chem. Soc.*, **82**, 5771 (1960).
9. C.L. Rollinson and R.C. White, *Inorg. Chem.*, **1**, 281 (1962).
10. C.W. Sink and A.B. Harvey, *J. Chem. Soc. (D)*, 1023 (1969).