

NOTES

Some Molecular Adducts of Telluracyclopentane-1,1-diiodide

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Some molecular adducts of telluracyclopentane-1,1-diiodide, $(\text{CH}_2)_4\text{TeI}_2$ of the type $(\text{CH}_2)_4\text{TeI}_2\cdot\text{L}$ (where L = pyridine-N-oxide, 4-picoline-N-oxide, 2,6-lutidine-N-oxide, triphenylphosphine oxide and tetramethylene sulphoxide) have been prepared and characterised by elemental analysis, conductivity, IR and X-ray photoelectron spectral data.

Although a large number of organotellurium(IV) compounds are reported¹, the telluracyclopentane-1,1-diiodide compounds are relatively less known². This paper describes the synthesis and the characterisation of telluracyclopentane-1,1-diiodide molecular adducts.

Telluracyclopentane-1,1-diiodide was prepared, purified and characterised according to literature procedure³.

1 Mmol of telluracyclopentane-1,1-diiodide dissolved in excess of acetone (20 ml) was refluxed for 3–4 hrs. with 2 mmol of pyridine-N-oxide (PyO), 4-picoline-N-oxide (4PicO), 2,6-lutidine-N-oxide, (LtO), triphenylphosphine oxide or tetramethylene sulphoxide. The solution was then distilled off and 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.

The X-ray photoelectron spectra were recorded on a VG Scientific ESCA-MK-II electron spectrometer. The MgK_α X-ray line (1253.6 eV) was used for photoexcitation. The $\text{Cu } ^2\text{p}_{3/2}$ (BE = 932.8 ± 0.2) and $\text{Au } ^4\text{f}_{7/2}$ (BE = 83.8 ± 0.1 eV) lines were used to calibrate the instrument and $\text{Ag } ^3\text{d}_{5/2}$ (BE = 368.2 eV) was used for cross-checking⁴.

All these molecular adducts are stable with high melting points. Elemental analyses were ±0.5% for C, H, N and I. The molar conductance data of below than 60 ohm^{-1} in DMF suggest that all these adducts are nonelectrolyte⁵ with composition of $(\text{CH}_2)_4\text{TeI}_2\cdot 2\text{L}$.

The IR $\nu_{\text{str. N-O}}$ in free bases (PyO, LtO and 4-PicO) around 1265–1245 cm^{-1} , shifted to 1210–1215 cm^{-1} on complexation^{6–8}. This decrease in the frequency of $\nu_{\text{N-O}}$ in the molecular adducts as compared to the free base is attributed to the coordination of the oxygen atom of the bases to the tellurium metal causing a decrease in π -character of the N–O bond^{9–11}. The IR $\nu_{\text{str. P-O}}$ vibration in free base triphenylphosphine oxide around 1192 cm^{-1} , moves to a lower frequency in molecular adduct^{12, 13}, showing coordination of triphenyl-

phosphine oxide to the tellurium metal ion through its oxygen atom. The IR $\nu_{\text{str. S-O}}$ vibration in free base tetramethylene sulphoxide around 1020 cm^{-1} ,¹⁴ disappears in molecular adducts and a new band with enhanced intensity appears at $970\text{--}915\text{ cm}^{-1}$ region, showing coordination of tellurium metal ion through its oxygen atom¹⁵. The bands at 320 cm^{-1} and $560\text{--}540\text{ cm}^{-1}$ in all these molecular adducts may be assigned to $\nu_{\text{Te-O}}$ ¹⁶ and $\nu_{\text{Te-C}}$ ¹⁷ respectively.

The binding energy data of $\text{Te } 3d_{3/2}$ and $\text{Te } 3d_{5/2}$ photoelectron peaks for $(\text{CH}_2)_4\text{TeI}_2$ were observed at 583.4 eV and 573.2 eV respectively, higher than their molecular adducts, $(\text{CH}_2)_4\text{TeI}_2 \cdot 2\text{L}$, 582.8 eV and 572.8 eV . From these XPS data one can conclude that all these bases are coordinated to tellurium metal ion¹⁸.

On the basis of the above data a structure $(\text{CH}_2)_4\text{TeI}_2 \leftarrow 2\text{L}$ is proposed to all these molecular adducts.

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