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# Structural Researches on Nitratocomplexes of Organotin. The Crystal Structure of the Triclinic Modification of Nitratotriphenyl(pyridine N-oxide)tin(IV)

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From the reaction of pyridine N-oxide with nitratotriphenyltin in dry acetone under nitrogen two types of crystal, belonging to the monoclinic and triclinic system, respectively, and having the same $[Sn(C_6H_5)_3(NO_3)(C_5H_5NO)]$ stoichiometry, have been obtained in the same preparation. We have already studied the structural properties of the monoclinic compound [1] as a part of a systematic investigation on adducts of nitratocomplexes of organotin with unidentate ligands containing oxygen groups such as N–O, S–O, P–O, As–O [1–5]. The present paper deals with the X-ray analysis of the triclinic modification, which was undertaken to permit a comparison of its molecular dimensions with those of the monoclinic compound.

## Experimental

The title compound occurs as colourless crystals with unit-cell dimensions: $a$ = 10.18(1), $b$ = 17.92(2), $c$ = 13.71(2) Å, $\alpha$ = 86.29(9), $\beta$ = 89.34(12), $\gamma$ = 60.73(7)°, $Z$ = 2, space group P$\bar{1}$. The intensities of 10507 independent reflections, 6937 of which labelled as "observed" having $I > 2\sigma(I)$, have been recorded up to 2σ = 56°, using the Mo-Kα radiation (λ = 0.7107 Å). X-ray diffraction measurements have been performed with a Siemens AED single-crystal diffractometer. The structure has been solved by Patterson and Fourier methods and refined by block-diagonal least-squares with anisotropic thermal parameters for the non-hydrogen atoms. The hydrogen atoms have been included in their calculated positions as fixed contributions to $F_c$ assuming isotropic thermal parameters. The final R was 5.2%.

TABLE I. Comparison of Bond Distances (Å) and Angles (°) at Tin in the Three Molecules.

| | A | B | M |
|---|---|---|---|
| Sn–C(1) | 2.116(10) | 2.075(9) | 2.096(9) |
| Sn–C(7) | 2.087(11) | 2.104(7) | 2.126(9) |
| Sn–C(13) | 2.084(8) | 2.142(11) | 2.112(9) |
| Sn–O(1) | 2.227(8) | 2.253(7) | 2.245(8) |
| Sn–O(4) | 2.299(7) | 2.238(6) | 2.227(8) |
| C(1)–Sn–C(7) | 112.7(10) | 128.9(10) | 118.0(3) |
| C(1)–Sn–C(13) | 126.3(10) | 112.6(9) | 120.8(3) |
| C(7)–Sn–C(13) | 120.9(10) | 118.4(9) | 120.9(3) |
| O(1)–Sn–C(1) | 82.8(5) | 96.4(6) | 97.3(3) |
| O(4)–Sn C(1) | 91.4(5) | 89.3(5) | 89.4(3) |
| O(1)–Sn–C(7) | 98.5(7) | 91.9(5) | 82.6(3) |
| O(4)–Sn–C(7) | 89.8(4) | 85.4(4) | 89.6(3) |
| O(1)–Sn–C(13) | 92.6(5) | 84.7(5) | 94.6(3) |
| O(4)–Sn–C(13) | 85.5(4) | 92.0(6) | 86.5(3) |
| O(1)–Sn–O(4) | 171.2(4) | 174.2(4) | 171.4(2) |

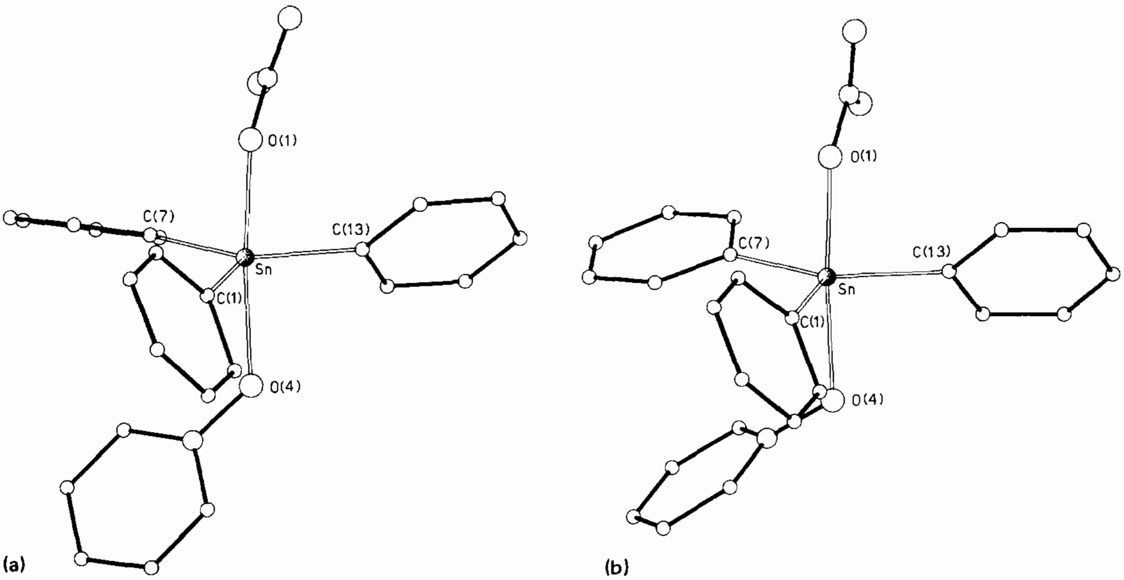

Fig. 1. A perspective view of the two independent molecules in the triclinic modification.

TABLE II. Comparison of the Main Dihedral Angles (°) in the Coordination Polyhedron of the Three Molecules.

| | | A | B | M |
|---|---|---|---|---|
| C(1)O(1)O(4)Sn | C(7)O(1)O(4)Sn | 111.4 | 127.5 | 119.0 |
| C(1)O(1)O(4)Sn | C(13)O(1)O(4)Sn | 131.2 | 111.5 | 117.9 |
| C(7)O(1)O(4)Sn | C(13)O(1)O(4)Sn | 117.4 | 121.0 | 123.1 |
| C(1)C(7)C(13)Sn | Sn–O(1) | 80.5 | 82.9 | 81.1 |
| C(1)C(7)C(13)Sn | Sn–O(4) | 86.5 | 86.1 | 88.3 |
| C(1)C(7)C(13)Sn | C(1)C(2)C(3)C(4)C(5)C(6) | 55.0 | 14.8 | 5.3 |
| C(1)C(7)C(13)Sn | C(7)C(8)C(9)C(10)C(11)C(12) | 19.4 | 56.1 | 33.3 |
| C(1)C(7)C(13)Sn | C(13)C(14)C(15)C(16)C(17)C(18) | 53.3 | 54.1 | 61.4 |
| C(1)C(7)C(13)Sn | O(1)O(2)O(3)N(1) | 71.5 | 84.2 | 78.7 |
| C(1)C(7)C(13)Sn | O(4)N(2)C(19)C(20)C(21)C(22)C(23) | 51.7 | 35.2 | 36.0 |

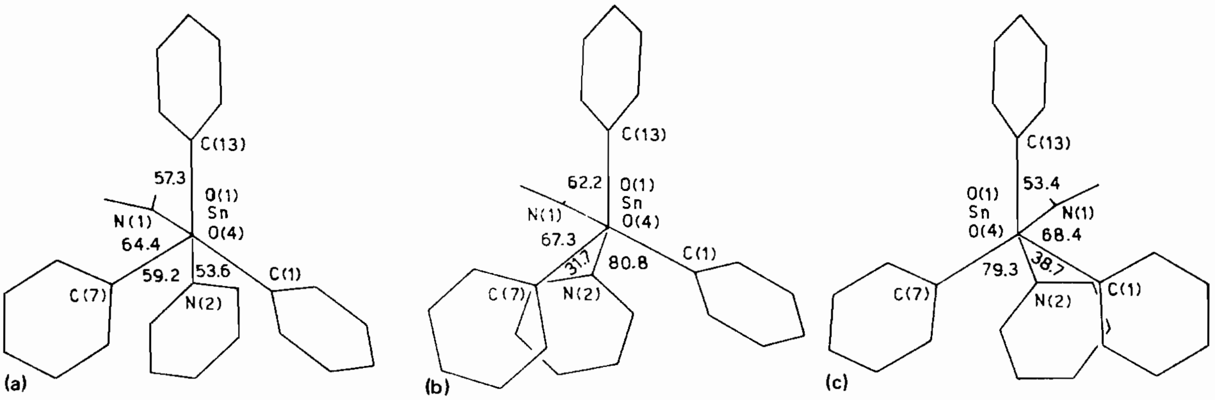

Fig. 2. Schemes showing the differences in the orientations of the ligands in the three molecules (torsional angles in degrees).

## Results and Discussion

In the asymmetric unit two independent molecules (A and B) are present, a perspective view of which is given in Fig. 1.

In Table I bond distances and angles in the coordination polyhedron for the molecules A and B of the present triclinic structure and for the molecule of the monoclinic structure (M) are compared. As can be seen, in most cases the agreement between corresponding values is rather good. The influence of packing in the conformation of the three molecules is shown in Table II and in the schemes of Figure 2, from which it can be observed that the coordination polyhedron, a slightly distorted trigonal bipyramid, is the same in the three types of molecules, while the orientations of the planes of the ligand molecules are different.

The structural parameters involving the pyridine N-oxide, the unidentate nitrate group and the phenyl rings are quite normal, and in good agreement with those observed in related compounds.

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