

SbCl₃, BiCl₃ and Na⁺ Complexes of Maleonitrile-Dithiacrown Ethers: Synthesis, Crystal Structures and DEP-MS Experiments

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Maleonitrile-Dithiacrown Ether Complexes, Antimony and Bismuth Trichlorides, Sodium,
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The reactions of MCl₃ (M = Sb, Bi) with maleonitrile-dithia-15-crown-5 (mn-15S₂O₃) and maleonitrile-dithia-18-crown-6 (mn-18S₂O₄) in MeCN yielded the complexes [MCl₃(mn-15S₂O₃)] {M = Sb(**1**), Bi(**2**)} and [MCl₃(mn-18S₂O₄)] {M = Sb(**3**), Bi(**4**)}, respectively. The pyramidal MCl₃ units are coordinated very weakly to the three oxygen and two sulphur donor atoms of mn-15S₂O₃ in **1** and **2**, and to the four oxygen donor atoms of mn-18S₂O₄ in **3** and **4**. Both mn-15S₂O₃ complexes, **1** and **2**, crystallize isotypically in the monoclinic space group *P*2(1)/*n* with four formula units per unit cell, while the isotypic mn-18S₂O₄ complexes, **3** and **4**, are triclinic, space group *P*-1, with two formula units per unit cell. In the SbCl₃ complexes, **1** and **3**, the mean contact distances between the Sb centres and the macrocyclic donor atoms are longer than the corresponding distances in their isostructural BiCl₃ analogues, **2** and **4**, which may reflect a stereochemical activity of the Sb^{III} lone pair. Under the conditions of DEP-MS experiments with **1** and **3** the monocationic SbCl₂⁺ complexes [SbCl₂(L)]⁺ (L = mn-15S₂O₃, mn-18S₂O₄) were detected.

NaSbCl₆ and mn-18S₂O₄ in MeCN furnished the 2:1 complex [Na(mn-18S₂O₄)₂]SbCl₆ {(**5**)SbCl₆}. In the complex cation **5** the sodium atom is coordinated sandwich-like through the eight oxygen atoms of two mn-18S₂O₄ molecules. Compound (**5**)SbCl₆ crystallize in the triclinic space group *P*-1 with two formula units per unit cell.

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