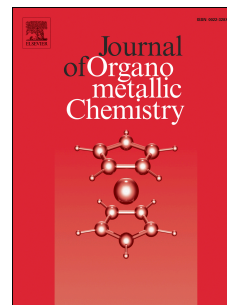


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Systematics of Boron Halide Complexes with Dichalcogenoether Ligands – Synthesis, Structures and Reaction Chemistry

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Abstract

The complexes $[BX_3(\mu\text{-L-L})BX_3]$ ($X = \text{Cl, Br, I}$; $\text{L-L} = \text{EtS}(\text{CH}_2)_2\text{SEt}$, $\text{MeSe}(\text{CH}_2)_2\text{SeMe}$), $[BX'_3\{o\text{-C}_6\text{H}_4(\text{SMe})_2\}]$ ($X' = \text{Cl, I}$), $[(\text{BBr}_3)_2\{o\text{-C}_6\text{H}_4(\text{SMe})_2\}]$, $[(\text{BBr}_3)_2\{o\text{-C}_6\text{H}_4(\text{SeMe})_2\}]$ and $[\text{BI}_3\{o\text{-C}_6\text{H}_4(\text{SeMe})_2\}]$ have been prepared as moisture-sensitive pale solids by reaction of the appropriate BX_3 with the dichalcogenoether in anhydrous n-hexane solution, and characterised by microanalysis, IR and multinuclear (^1H , ^{11}B , $^{77}\text{Se}\{^1\text{H}\}$) NMR spectroscopy. In contrast, the $[\text{BF}_3(\mu\text{-L-L})\text{BF}_3]$, $[(\text{BF}_3)_2\{o\text{-C}_6\text{H}_4(\text{SMe})_2\}]$ and $[(\text{BF}_3)_2\{o\text{-C}_6\text{H}_4(\text{SeMe})_2\}]$, made from BF_3 and the neat ligands, are viscous oils which have a significant vapour pressure of BF_3 at ambient temperatures. X-ray crystal structures are reported for $[\text{BX}_3\{\mu\text{-EtS}(\text{CH}_2)_2\text{SEt}\}\text{BX}_3]$ ($X = \text{Cl, Br, I}$), $[\text{BBr}_3\{\mu\text{-MeSe}(\text{CH}_2)_2\text{SeMe}\}\text{BBr}_3]$, $[\text{BCl}_3\{o\text{-C}_6\text{H}_4(\text{SMe})_2\}]$ and $[(\text{BBr}_3)_2\{\mu\text{-}o\text{-C}_6\text{H}_4(\text{SeMe})_2\}]$. The complexes $[(\text{BX}_3)_2\{\text{MeTe}(\text{CH}_2)_3\text{TeMe}\}]$ ($X = \text{F, Cl, Br}$) have been identified in solution by multinuclear NMR spectroscopy, but decompose rapidly, whilst $o\text{-C}_6\text{H}_4(\text{TeMe})_2$ decomposes immediately on contact with BBr_3 or BCl_3 . Dealkylation of some of the chalcogenoether ligands at room temperature by BI_3 , to yield complexes including $[\text{BI}_2\{o\text{-C}_6\text{H}_4\text{S}(\text{SMe})\}]$ and $[\text{BI}_2\{o\text{-C}_6\text{H}_4\text{Se}(\text{SeMe})\}]$, has been identified and the X-ray structure of $[\text{BI}_2\{o\text{-C}_6\text{H}_4\text{Se}(\text{SeMe})\}]$ determined. The trends in behaviour along the series of boron halides and with the various chalcogenoethers are described and compared with the behaviour of BX_3 with neutral phosphorus and arsenic donor ligands (Burt *et. al.*, *Inorg. Chem.*, 2016, **55**, 8852) and with $[\text{BX}_3(\text{EMe}_2)]$ ($\text{E} = \text{S, Se, Te}$) (Okio *et. al.*, *J. Organometal. Chem.*, 2017, **848**, 232).

Introduction

Almost 100 years ago, G. N. Lewis proposed the concept of (Lewis) acids and bases as electron pair acceptors and electron pair donors respectively [1], and this concept has become a classification widely used in much p-block chemistry. Boron and aluminium trihalides are textbook examples of Lewis acids. Study of boron derivatives BY_3 , where $\text{Y} = \text{halide, H, substituted aryl}$, remains an active area of research both in terms of fundamental chemistry [2-5], and their many applications in organic synthesis [6,7]. Trends in Lewis acidity with Y are commonly quoted in textbooks, for

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