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## Complexes of germanium bromides with 4-picoline and 3,4-lutidine

Michael Bolte,<sup>a\*</sup> Karl Hensen<sup>b</sup> and Alexander Faber<sup>b</sup>

<sup>a</sup>Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and <sup>b</sup>Institut für Physikalische und Theoretische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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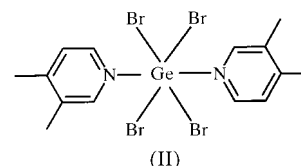
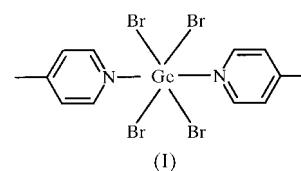
Data validation number: IUC0000284

The reactions of  $\text{GeBr}_4$  with 4-methylpyridine and 3,4-dimethylpyridine lead to tetrabromobis(4-methylpyridine)-germanium,  $[\text{GeBr}_4(\text{C}_6\text{H}_7\text{N})_2]$ , and tetrabromobis(3,4-dimethylpyridine)germanium,  $[\text{GeBr}_4(\text{C}_7\text{H}_9\text{N})_2]$ , respectively. These structures show the same features as the corresponding silicon complexes.

### Comment

In recent years, the extension of the coordination sphere of silicon in complexes with organic nitrogen bases has been the subject of numerous studies (Bechstein *et al.*, 1990; Chuit *et al.*, 1993; Kane *et al.*, 1998; Hensen *et al.*, 1998; Hensen, Mayr-Stein, Spangenberg, Bolte & Rühl, 2000). Several complexes of silicon halides are already known, but little is known about adducts of germanium halides (Adley *et al.*, 1972; Bolte *et al.*, 2000) with aromatic nitrogen bases.

We have already published the structure of the addition compound of  $\text{GeBr}_4$  with 3,4-dimethylpyridine (Hensen *et al.*, 1999) and we present here two further structures of this kind. Tetrabromobis(4-methylpyridine)germanium, (I), is isomorphous with tetrabromobis(4-methylpyridine)silicon (Hensen, Mayr-Stein, Spangenberg & Bolte, 2000) and tetrachlorobis(4-methylpyridine)silicon (Bolte *et al.*, 2000), and tetrabromobis(3,4-dimethylpyridine)germanium, (II), is isomorphous with tetrabromobis(3,4-dimethylpyridine)silicon (Hensen, Mayr-Stein, Stumpf *et al.*, 2000); the molecular geometry of this type of compound has been extensively discussed. As a result of this, we refrain from repeating the discussion of these structures. The present results demonstrate that the reaction of  $\text{GeBr}_4$  with aromatic nitrogen bases leads to the same results as the reaction of silicon halides with aromatic nitrogen bases and, furthermore, using  $\text{GeCl}_4$  instead of  $\text{GeBr}_4$  yields analogous results.



### Experimental

Both title compounds were prepared as described by Faber (2000). Single crystals were obtained from chloroform solutions.

#### Compound (I)

##### Crystal data

$[\text{GeBr}_4(\text{C}_6\text{H}_7\text{N})_2]$   
 $M_r = 578.48$   
 Orthorhombic,  $Pmna$   
 $a = 7.360$  (1) Å  
 $b = 8.360$  (1) Å  
 $c = 13.561$  (1) Å  
 $V = 834.40$  (16) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 2.302$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 5051 reflections  
 $\theta = 1-25^\circ$   
 $\mu = 11.401$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Needle, yellow  
 $0.36 \times 0.16 \times 0.10$  mm

##### Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.089$ ,  $T_{\max} = 0.319$   
 14 794 measured reflections  
 1122 independent reflections  
 869 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.0744$   
 $\theta_{\text{max}} = 28.27^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -11 \rightarrow 11$   
 $l = -18 \rightarrow 18$   
 112 standard reflections  
 frequency: 900 min  
 intensity decay: none

##### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.056$   
 $S = 1.075$   
 1122 reflections  
 57 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 1.2129P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.48$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.64$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.0027 (3)

#### Compound (II)

##### Crystal data

$[\text{GeBr}_4(\text{C}_7\text{H}_9\text{N})_2]$   
 $M_r = 606.53$   
 Orthorhombic,  $Cmca$   
 $a = 7.477$  (1) Å  
 $b = 17.942$  (5) Å  
 $c = 13.194$  (3) Å  
 $V = 1770.0$  (7) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 2.276$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 3846 reflections  
 $\theta = 1-25^\circ$   
 $\mu = 10.755$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Plate, yellow  
 $0.22 \times 0.12 \times 0.05$  mm

## Data collection

Siemens CCD three-circle diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.200$ ,  $T_{\max} = 0.580$   
 12 371 measured reflections  
 1134 independent reflections  
 898 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.099$   
 $\theta_{\text{max}} = 27.84^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -23 \rightarrow 23$   
 $l = -17 \rightarrow 17$   
 122 standard reflections  
 frequency: 1200 min  
 intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.077$   
 $S = 1.070$   
 1134 reflections  
 62 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 6.1521P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.62 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.78 \text{ e } \text{\AA}^{-3}$

All H atoms were located by difference Fourier synthesis and the H atoms bonded to carbon were refined with fixed individual displacement parameters [ $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ ] using a riding model with aromatic C–H = 0.95 Å or methyl C–H = 0.98 Å. The H atoms of the methyl groups in (I) are disordered. Two orientations differing by a 60° rotation about the C<sub>Ph</sub>–C<sub>Me</sub> bond could be identified.

For both compounds, data collection: SMART (Siemens, 1995); cell refinement: SMART; data reduction: SAINT (Siemens, 1995);

program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); software used to prepare material for publication: SHELXL97.

## References

- Adley, A. D., Bird, P. H., Fraser, A. R. & Onyszchuk, M. (1972). *Inorg. Chem.* **11**, 1402–1409.
- Bechstein, O., Ziemer, B., Hass, D., Troyanov, S. I., Rybakov, V. B. & Maso, G. N. (1990). *Z. Anorg. Allg. Chem.* **582**, 211–216.
- Bolte, M., Hensen, K. & Faber, A. (2000). *Acta Cryst.* **C56**, e497–498.
- Chuit, C., Corriu, R. J. P., Reye, C. & Young, J. C. (1993). *Chem. Rev.* **93**, 1371–1448.
- Faber, A. (2000). PhD thesis, University of Frankfurt, Germany.
- Hensen, K., Faber, A. & Bolte, M. (1999). *Acta Cryst.* **C55**, 1774–1775.
- Hensen, K., Mayr-Stein, R., Spangenberg, B. & Bolte, M. (2000). *Acta Cryst.* **C56**, 610–613.
- Hensen, K., Mayr-Stein, R., Spangenberg, B., Bolte, M. & Rühl, St. (2000). *Z. Naturforsch. Teil B*, **55**, 248–252.
- Hensen, K., Mayr-Stein, R., Stumpf, T., Pickel, P., Bolte, M. & Fleischer, H. (2000). *J. Chem. Soc. Dalton Trans.* pp. 473–477.
- Hensen, K., Stumpf, T., Bolte, M., Näther, C. & Fleischer, H. (1998). *J. Am. Chem. Soc.* **120**, 10402–10408.
- Kane, K. M., Lorenz, C. R., Heilman, D. M. & Lemke, F. R. (1998). *Inorg. Chem.* **37**, 669–673.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Siemens (1995). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.