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XRD data

The structure of **126** was obtained from an X-ray crystallographic analysis. The APEX2 crystallographic software on the Bruker Smart X2S provided a carbon framework with essentially the expected connectivity. Refinement of the data with Crystals²³⁵⁻²³⁶ (Version 14.11) provided a structure that agreed with the expected result. The crystallographic data is given below.

Table 6.1: Crystal data and structure refinement for 126

| | | | |
|-------------------|---|-----------------------------------|-----------|
| Empirical formula | C ₁₈ H ₁₃ Cl ₆ O | V (Å ³) | 1686.6(7) |
| Molecular weight | 432.99 | Z | 4 |
| Crystal size | 0.05 mm × 0.05 mm × 1.00 mm | μ (mm ⁻¹) | 1.018 |
| Crystal system | Orthorhombic | ρ calc (mg·cm ⁻³) | 1686.5(7) |
| Space group | P2 ₁ 2 ₁ 2 ₁ | 2θ (°) | 25.04 |
| T (K) | 300(2) | Reflections collected | 10805 |
| a (Å) | 7.7317(16) | Goodness-of-fit on F ² | 0.971 |
| b (Å) | 9.499(2) | R-factor | 0.051 |
| c (Å) | 22.963(5) | R ₁ | 0.0525 |
| α = β = γ (°) | 90 | - | - |

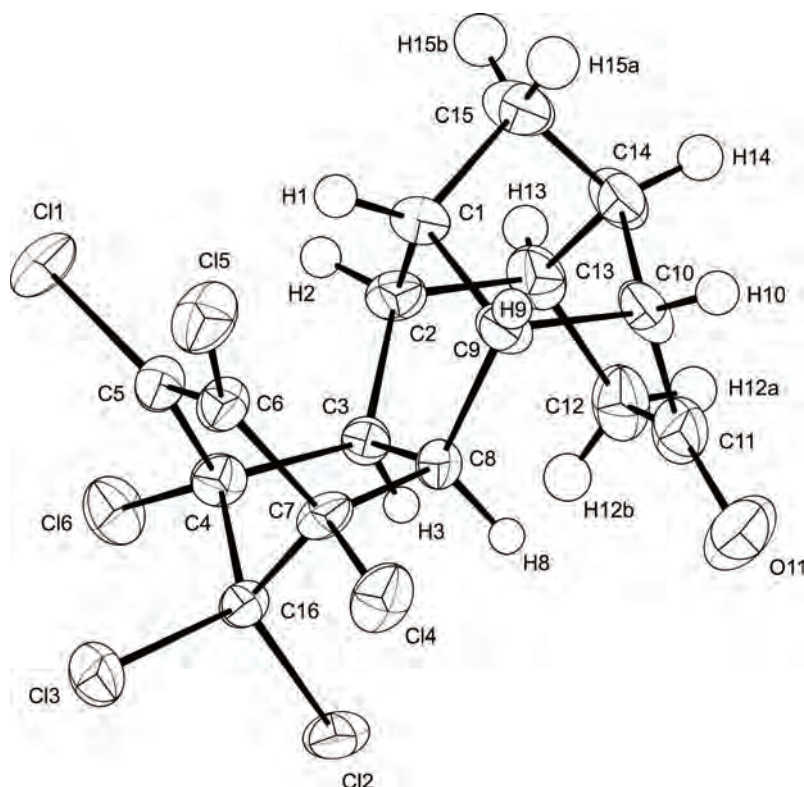


Table 6.2: Distances for structure 126b

| | | | | | |
|-----|------|-----------|-----|------|-----------|
| C1 | C2 | 1.549(6)Å | C1 | C9 | 1.539(6)Å |
| C1 | C15 | 1.529(6)Å | C1 | H1 | 0.987Å |
| C2 | C3 | 1.522(6)Å | C2 | C13 | 1.592(6)Å |
| C2 | H2 | 0.993Å | C3 | C4 | 1.560(6)Å |
| C3 | C8 | 1.570(5)Å | C3 | H3 | 0.969Å |
| C4 | C5 | 1.517(6)Å | C4 | C16 | 1.579(6)Å |
| C4 | Cl6 | 1.772(4)Å | C5 | C6 | 1.317(6)Å |
| C5 | Cl1 | 1.698(4)Å | C6 | C7 | 1.521(5)Å |
| C6 | Cl5 | 1.700(4)Å | C7 | C8 | 1.560(5)Å |
| C7 | C16 | 1.549(6)Å | C7 | Cl4 | 1.761(4)Å |
| C8 | C9 | 1.534(5)Å | C8 | H8 | 0.987Å |
| C9 | C10 | 1.607(6)Å | C9 | H9 | 0.974Å |
| C10 | C11 | 1.508(7)Å | C10 | C14 | 1.536(7)Å |
| C10 | H10 | 0.982Å | C11 | C12 | 1.516(7)Å |
| C11 | O8 | 1.225(6)Å | C12 | C13 | 1.519(7)Å |
| C12 | H12b | 0.958Å | C12 | H12a | 0.970Å |
| C13 | C14 | 1.540(7)Å | C13 | H13 | 0.988Å |
| C14 | C15 | 1.510(7)Å | C14 | H14 | 0.987Å |
| C15 | H15b | 0.982Å | C15 | H15a | 0.957Å |
| C16 | Cl2 | 1.764(4)Å | C16 | Cl3 | 1.766(4)Å |

Table 6.3: Parameters for structure 126b

| Label | x | y | z | U _{iso/equiv} | Occupancy |
|-------|-------------|-------------|-------------|------------------------|-----------|
| C1 | 0.1534(6) | 0.5360(4) | 0.3545(2) | 0.0384 | 1.0000 |
| C2 | 0.1823(6) | 0.5873(5) | 0.41775(17) | 0.0349 | 1.0000 |
| C3 | 0.3563(5) | 0.6623(4) | 0.41774(16) | 0.0255 | 1.0000 |
| C4 | 0.3627(5) | 0.8210(5) | 0.40039(16) | 0.0298 | 1.0000 |
| C5 | 0.2667(5) | 0.8475(4) | 0.34384(18) | 0.0331 | 1.0000 |
| C6 | 0.3565(5) | 0.7876(4) | 0.30185(17) | 0.0313 | 1.0000 |
| C7 | 0.5165(5) | 0.7209(4) | 0.32878(15) | 0.0287 | 1.0000 |
| C8 | 0.4618(5) | 0.5926(4) | 0.36695(15) | 0.0267 | 1.0000 |
| C9 | 0.3374(5) | 0.4836(4) | 0.34096(18) | 0.0326 | 1.0000 |
| C10 | 0.3415(6) | 0.3380(5) | 0.37656(19) | 0.0389 | 1.0000 |
| C11 | 0.4604(6) | 0.3434(5) | 0.4286(2) | 0.0447 | 1.0000 |
| C12 | 0.3550(7) | 0.3974(5) | 0.4796(2) | 0.0470 | 1.0000 |
| C13 | 0.1842(7) | 0.4424(5) | 0.4526(2) | 0.0424 | 1.0000 |
| C14 | 0.1604(6) | 0.3327(5) | 0.4039(2) | 0.0464 | 1.0000 |
| C15 | 0.0437(7) | 0.4028(6) | 0.3599(3) | 0.0560 | 1.0000 |
| C16 | 0.5537(5) | 0.8322(4) | 0.37649(17) | 0.0310 | 1.0000 |
| Cl1 | 0.07123(15) | 0.92830(14) | 0.33910(6) | 0.0511 | 1.0000 |
| Cl2 | 0.71296(14) | 0.78278(12) | 0.42754(5) | 0.0422 | 1.0000 |
| Cl3 | 0.60831(15) | 1.00049(12) | 0.34951(6) | 0.0462 | 1.0000 |

| | | | | | |
|------|-------------|-------------|-------------|--------|--------|
| Cl4 | 0.68820(15) | 0.68574(12) | 0.28055(4) | 0.0430 | 1.0000 |
| Cl5 | 0.30279(17) | 0.77504(13) | 0.23027(4) | 0.0472 | 1.0000 |
| Cl6 | 0.30810(17) | 0.93950(12) | 0.45706(4) | 0.0416 | 1.0000 |
| O8 | 0.6146(5) | 0.3142(5) | 0.43011(18) | 0.0642 | 1.0000 |
| H1 | 0.1104(6) | 0.6119(4) | 0.3289(2) | 0.0456 | 1.0000 |
| H2 | 0.0900(6) | 0.6519(5) | 0.43152(17) | 0.0406 | 1.0000 |
| H3 | 0.4201(5) | 0.6482(4) | 0.45360(16) | 0.0321 | 1.0000 |
| H8 | 0.5682(5) | 0.5472(4) | 0.38157(15) | 0.0318 | 1.0000 |
| H9 | 0.3535(5) | 0.4673(4) | 0.29944(18) | 0.0393 | 1.0000 |
| H10 | 0.3696(6) | 0.2586(5) | 0.35084(19) | 0.0474 | 1.0000 |
| H12b | 0.4122(7) | 0.4752(5) | 0.4978(2) | 0.0555 | 1.0000 |
| H12a | 0.3422(7) | 0.3226(5) | 0.5080(2) | 0.0550 | 1.0000 |
| H13 | 0.0901(7) | 0.4404(5) | 0.4817(2) | 0.0505 | 1.0000 |
| H14 | 0.1278(6) | 0.2385(5) | 0.4183(2) | 0.0515 | 1.0000 |
| H15b | -0.0728(7) | 0.4213(6) | 0.3752(3) | 0.0680 | 1.0000 |
| H15a | 0.0318(7) | 0.3517(6) | 0.3241(3) | 0.0679 | 1.0000 |

Table 6.4: Angles for structure 126b

| | | | | | | | |
|-----|-----|-----|-----------|-----|-----|-----|-----------|
| C2 | C1 | C9 | 99.1(3)° | C2 | C1 | C15 | 105.3(4)° |
| C9 | C1 | C15 | 105.1(4)° | C2 | C1 | H1 | 112.167° |
| C9 | C1 | H1 | 115.331° | C15 | C1 | H1 | 117.781° |
| C1 | C2 | C3 | 105.9(3)° | C1 | C2 | C13 | 101.6(3)° |
| C3 | C2 | C13 | 113.3(4)° | C1 | C2 | H2 | 112.934° |
| C3 | C2 | H2 | 110.230° | C13 | C2 | H2 | 112.400° |
| C2 | C3 | C4 | 118.7(3)° | C2 | C3 | C8 | 105.2(3)° |
| C4 | C3 | C8 | 101.6(3)° | C2 | C3 | H3 | 112.655° |
| C4 | C3 | H3 | 109.541° | C8 | C3 | H3 | 107.959° |
| C3 | C4 | C5 | 111.3(3)° | C3 | C4 | C16 | 100.6(3)° |
| C5 | C4 | C16 | 98.6(3)° | C3 | C4 | C16 | 114.8(3)° |
| C5 | C4 | Cl6 | 114.0(3)° | C16 | C4 | C16 | 115.8(3)° |
| C4 | C5 | C6 | 107.3(4)° | C4 | C5 | Cl1 | 124.4(3)° |
| C6 | C5 | Cl1 | 128.1(3)° | C5 | C6 | C7 | 108.1(3)° |
| C5 | C6 | Cl5 | 127.5(3)° | C7 | C6 | Cl5 | 124.2(3)° |
| C6 | C7 | C8 | 109.5(3)° | C6 | C7 | C16 | 98.9(3)° |
| C8 | C7 | C16 | 100.7(3)° | C6 | C7 | Cl4 | 115.9(3)° |
| C8 | C7 | Cl4 | 114.1(3)° | C16 | C7 | Cl4 | 115.7(3)° |
| C3 | C8 | C7 | 103.2(3)° | C3 | C8 | C9 | 104.3(3)° |
| C7 | C8 | C9 | 118.6(3)° | C3 | C8 | H8 | 111.366° |
| C7 | C8 | H8 | 107.857° | C9 | C8 | H8 | 111.112° |
| C1 | C9 | C8 | 106.4(3)° | C1 | C9 | C10 | 101.2(3)° |
| C8 | C9 | C10 | 111.8(3)° | C1 | C9 | H9 | 111.474° |
| C8 | C9 | H9 | 114.100° | C10 | C9 | H9 | 111.028° |
| C9 | C10 | C11 | 112.7(3)° | C9 | C10 | C14 | 102.6(4)° |
| C11 | C10 | C14 | 103.5(4)° | C9 | C10 | H10 | 111.044° |

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| | | | |
|------|-----|------|-----------|
| C11 | C10 | H10 | 111.571° |
| C10 | C11 | C12 | 107.2(4)° |
| C12 | C11 | O8 | 125.3(5)° |
| C11 | C12 | H12b | 110.566° |
| C11 | C12 | H12a | 109.030° |
| H12b | C12 | H12a | 108.495° |
| C2 | C13 | C14 | 102.6(4)° |
| C2 | C13 | H13 | 110.498° |
| C14 | C13 | H13 | 112.918° |
| C10 | C14 | C15 | 104.9(4)° |
| C10 | C14 | H14 | 113.519° |
| C15 | C14 | H14 | 118.105° |
| C1 | C15 | H15b | 112.898° |
| C1 | C15 | H15a | 113.750° |
| H15b | C15 | H15a | 108.019° |
| C4 | C16 | Cl2 | 113.8(3)° |
| C4 | C16 | Cl3 | 114.0(3)° |
| Cl2 | C16 | Cl3 | 107.9(2)° |

| | | | |
|-----|-----|------|-----------|
| C14 | C10 | H10 | 115.017° |
| C10 | C11 | O8 | 127.5(5)° |
| C11 | C12 | C13 | 104.4(4)° |
| C13 | C12 | H12b | 111.271° |
| C13 | C12 | H12a | 113.062° |
| C2 | C13 | C12 | 117.1(4)° |
| C12 | C13 | C14 | 102.1(4)° |
| C12 | C13 | H13 | 111.077° |
| C10 | C14 | C13 | 99.6(4)° |
| C13 | C14 | C15 | 105.1(4)° |
| C13 | C14 | H14 | 113.679° |
| C1 | C15 | C14 | 95.0(4)° |
| C14 | C15 | H15b | 112.779° |
| C14 | C15 | H15a | 114.097° |
| C4 | C16 | C7 | 91.5(3)° |
| C7 | C16 | Cl2 | 114.7(3)° |
| C7 | C16 | Cl3 | 114.5(3)° |
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