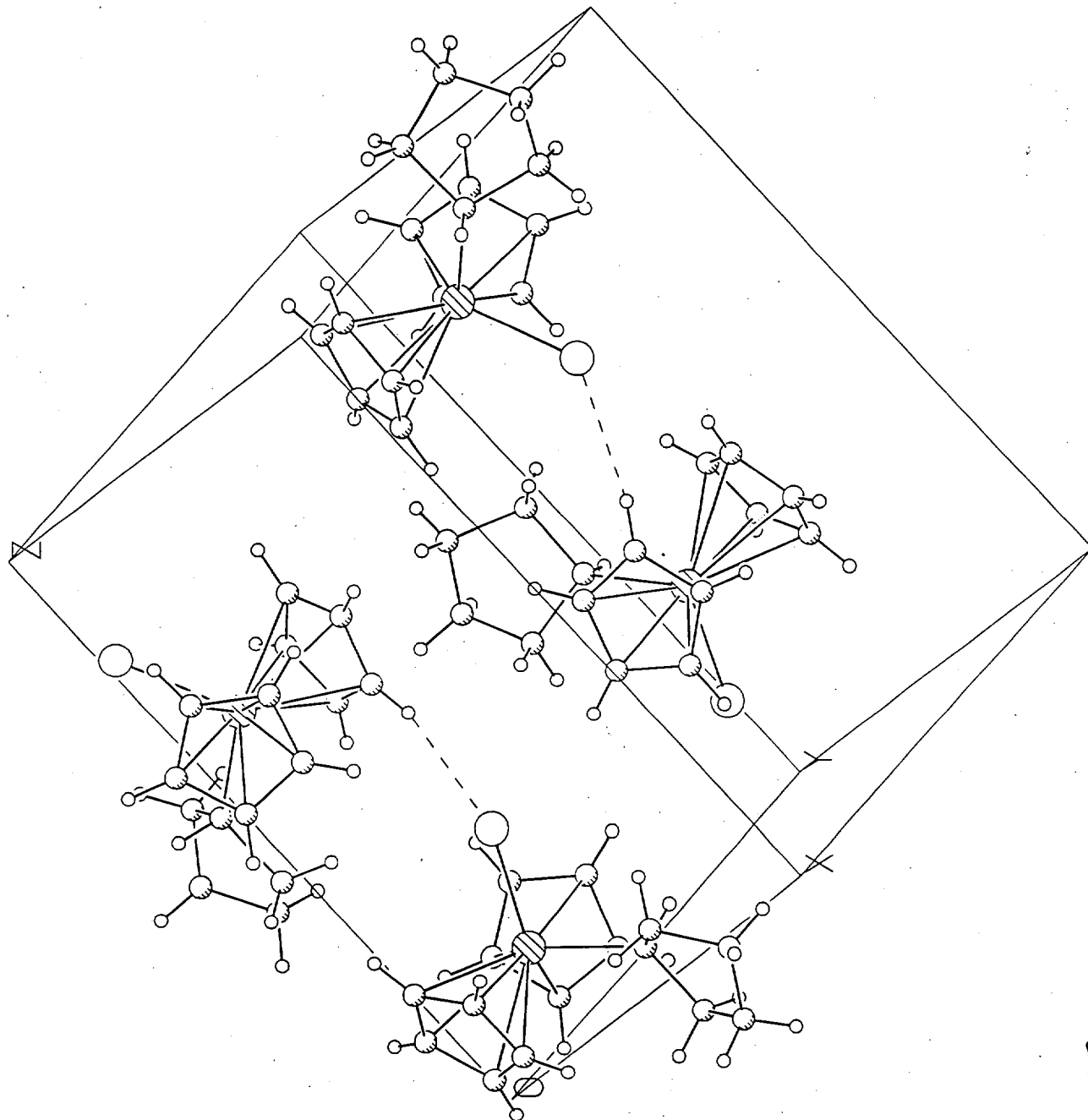


Table 1. Crystal data and structure refinement for 8

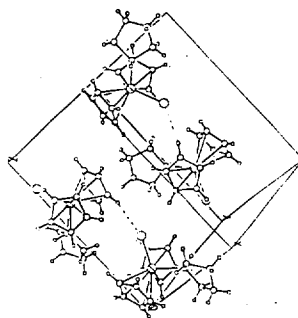
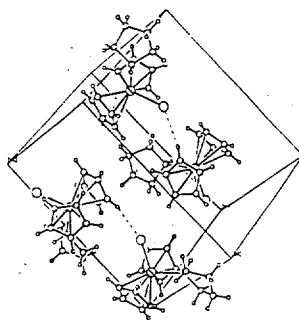
| | |
|-------------------------|---------------------------------------|
| Empirical formula | C ₁₅ H ₁₉ Cl Zr |
| Formula weight | 325.97 |
| Crystallization Solvent | Diethyl ether |
| Crystal Habit | Blade |
| Crystal size | 0.07 x 0.07 x 0.37 mm ³ |
| Crystal color | Pale yellow |

Data Collection

| | | |
|--|--|--|
| Preliminary Photos | None | |
| Type of diffractometer | CAD-4 | |
| Wavelength | 0.71073 Å MoK α | |
| Data Collection Temperature | 85 K | |
| θ range for reflections used in lattice determination | 13.5 to 14.3° | |
| Unit cell dimensions | a = 8.860(5) Å b = 11.551(3) Å c = 13.231(10) Å | $\alpha = 90^\circ$ $\beta = 90^\circ$ $\gamma = 90^\circ$ |
| Volume | 1354.1(13) Å ³ | |
| Z | 4 | |
| Crystal system | Orthorhombic | |
| Space group | P2 ₁ 2 ₁ 2 ₁ | |
| Density (calculated) | 1.599 Mg/m ³ | |
| F(000) | 664 | |
| θ range for data collection | 2.34 to 24.97° | |
| Completeness to $\theta = 24.97^\circ$ | 99.9 % | |
| Index ranges | 0 \leq h \leq 10, -13 \leq k \leq 13, -15 \leq l \leq 15 | |
| Data collection scan type | ω scans | |
| Reflections collected | 5917 | |
| Independent reflections | 1386 [R _{int} = 0.017; GOF _{merge} = 1.20] | |
| Absorption coefficient | 0.984 mm ⁻¹ | |
| Absorption correction | None | |
| Number of standards | 3 reflections measured every 75 min. | |
| Variation of standards | 1.06%. | |



Unit cell contents of **3**
showing the unit cell boundaries.



Stereo view of the unit cell boundaries for **8** showing the cell boundaries.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **8. $U(\text{eq})$ is defined as the trace of the orthogonalized U^i tensor.**

| | x | y | z | U_{eq} |
|-------|----------|----------|---------|-----------------|
| Zr | 1975(1) | 9852(1) | 6492(1) | 9(1) |
| Cl | 569(1) | 9790(1) | 8074(1) | 17(1) |
| C(1) | 2110(6) | 7650(4) | 6545(4) | 18(1) |
| C(2) | 2437(6) | 7978(4) | 5540(4) | 15(1) |
| C(3) | 3814(6) | 8582(5) | 5559(5) | 17(1) |
| C(4) | 4324(6) | 8637(4) | 6556(5) | 22(1) |
| C(5) | 3273(8) | 8050(4) | 7157(4) | 19(1) |
| C(6) | 1557(6) | 12022(4) | 6569(4) | 13(1) |
| C(7) | 1877(7) | 11745(4) | 5567(4) | 14(1) |
| C(8) | 3352(6) | 11355(4) | 5504(4) | 16(1) |
| C(9) | 3982(6) | 11375(4) | 6499(5) | 17(1) |
| C(10) | 2848(7) | 11784(4) | 7150(4) | 17(1) |
| C(11) | -123(5) | 9650(4) | 5515(3) | 12(1) |
| C(12) | -68(5) | 9625(4) | 4327(3) | 13(1) |
| C(13) | -1482(5) | 10277(5) | 3967(4) | 18(1) |
| C(14) | -2540(5) | 10272(5) | 4874(3) | 15(1) |
| C(15) | -1479(5) | 10443(4) | 5753(4) | 15(1) |

Table 3. Selected bond lengths [Å] and angles [°] for \mathcal{B}

| | | | |
|--------------------|------------|------------------|------------|
| Zr-Cent(1) | 2.2305(7) | Zr-Pln(1) | 2.2302(29) |
| Zr-Cent(2) | 2.2094(7) | Zr-Pln(2) | 2.2093(27) |
| Zr-C(11) | 2.276(5) | | |
| Zr-Cl | 2.4373(18) | | |
| Cent(1)-Zr-Cent(2) | 130.83(3) | Pln(1)-Zr-Pln(2) | 128.61(15) |
| C(11)-Zr-Cl | 93.85(13) | | |

Cent(1) is the centroid formed by C(1), C(2), C(3), C(4) and C(5).

Cent(2) is the centroid formed by C(6), C(7), C(8), C(9) and C(10).

Pln(1) is the plane formed by C(1), C(2), C(3), C(4) and C(5).

Pln(2) is the plane formed by C(6), C(7), C(8), C(9) and C(10).

Table 4. Bond lengths [Å] and angles [°] for β

| | | | |
|--------------------|------------|----------------|------------|
| Zr-Cent(1) | 2.2305(7) | C(11)-Zr-Cl | 93.85(13) |
| Zr-Cent(2) | 2.2094(7) | C(11)-Zr-C(8) | 99.97(19) |
| Zr-Pln(1) | 2.2302(29) | Cl-Zr-C(8) | 136.13(13) |
| Zr-Pln(2) | 2.2093(27) | C(11)-Zr-C(9) | 130.89(19) |
| Zr-C(11) | 2.276(5) | Cl-Zr-C(9) | 112.36(15) |
| Zr-Cl | 2.4373(18) | C(8)-Zr-C(9) | 33.3(2) |
| Zr-C(8) | 2.493(5) | C(11)-Zr-C(7) | 77.53(19) |
| Zr-C(9) | 2.501(5) | Cl-Zr-C(7) | 115.26(13) |
| Zr-C(7) | 2.507(5) | C(8)-Zr-C(7) | 32.14(18) |
| Zr-C(4) | 2.511(5) | C(9)-Zr-C(7) | 54.04(19) |
| Zr-C(3) | 2.516(6) | C(11)-Zr-C(4) | 129.73(18) |
| Zr-C(10) | 2.518(5) | Cl-Zr-C(4) | 112.22(17) |
| Zr-C(5) | 2.535(5) | C(8)-Zr-C(4) | 90.08(19) |
| Zr-C(2) | 2.537(5) | C(9)-Zr-C(4) | 78.69(17) |
| Zr-C(6) | 2.537(5) | C(7)-Zr-C(4) | 122.2(2) |
| Zr-C(1) | 2.546(4) | C(11)-Zr-C(3) | 100.99(19) |
| C(1)-C(5) | 1.390(8) | Cl-Zr-C(3) | 137.36(14) |
| C(1)-C(2) | 1.414(8) | C(8)-Zr-C(3) | 80.32(18) |
| C(1)-H(1) | 0.9300 | C(9)-Zr-C(3) | 87.24(19) |
| C(2)-C(3) | 1.405(8) | C(7)-Zr-C(3) | 106.94(18) |
| C(2)-H(2) | 0.9300 | C(4)-Zr-C(3) | 32.3(2) |
| C(3)-C(4) | 1.397(9) | C(11)-Zr-C(10) | 122.54(19) |
| C(3)-H(3) | 0.9300 | Cl-Zr-C(10) | 83.45(14) |
| C(4)-C(5) | 1.400(9) | C(8)-Zr-C(10) | 54.06(19) |
| C(4)-H(4) | 0.9300 | C(9)-Zr-C(10) | 32.49(19) |
| C(5)-H(5) | 0.9300 | C(7)-Zr-C(10) | 53.55(17) |
| C(6)-C(7) | 1.393(8) | C(4)-Zr-C(10) | 103.2(2) |
| C(6)-C(10) | 1.405(8) | C(3)-Zr-C(10) | 119.18(19) |
| C(6)-H(6) | 0.9300 | C(11)-Zr-C(5) | 118.93(19) |
| C(7)-C(8) | 1.384(8) | Cl-Zr-C(5) | 84.81(14) |
| C(7)-H(7) | 0.9300 | C(8)-Zr-C(5) | 122.2(2) |
| C(8)-C(9) | 1.430(8) | C(9)-Zr-C(5) | 104.7(2) |
| C(8)-H(8) | 0.9300 | C(7)-Zr-C(5) | 154.3(2) |
| C(9)-C(10) | 1.404(8) | C(4)-Zr-C(5) | 32.2(2) |
| C(9)-H(9) | 0.9300 | C(3)-Zr-C(5) | 53.02(19) |
| C(10)-H(10) | 0.9300 | C(10)-Zr-C(5) | 117.92(18) |
| C(11)-C(15) | 1.543(7) | C(11)-Zr-C(2) | 76.27(18) |
| C(11)-C(12) | 1.573(6) | Cl-Zr-C(2) | 118.95(13) |
| C(11)-H(11) | 0.9800 | C(8)-Zr-C(2) | 104.78(18) |
| C(12)-C(13) | 1.537(7) | C(9)-Zr-C(2) | 119.17(18) |
| C(12)-H(12A) | 0.9700 | C(7)-Zr-C(2) | 120.49(16) |
| C(12)-H(12B) | 0.9700 | C(4)-Zr-C(2) | 53.62(19) |
| C(13)-C(14) | 1.523(6) | C(3)-Zr-C(2) | 32.29(17) |
| C(13)-H(13A) | 0.9700 | C(10)-Zr-C(2) | 151.4(2) |
| C(13)-H(13B) | 0.9700 | C(5)-Zr-C(2) | 53.08(18) |
| C(14)-C(15) | 1.508(6) | C(11)-Zr-C(6) | 90.27(17) |
| C(14)-H(14B) | 0.9700 | Cl-Zr-C(6) | 85.38(13) |
| C(14)-H(14C) | 0.9700 | C(8)-Zr-C(6) | 53.41(18) |
| C(15)-H(15C) | 0.9700 | C(9)-Zr-C(6) | 53.71(16) |
| C(15)-H(15A) | 0.9700 | C(7)-Zr-C(6) | 32.05(17) |
| | | C(4)-Zr-C(6) | 132.20(17) |
| Cent(1)-Zr-Cent(2) | 130.83(3) | C(3)-Zr-C(6) | 133.68(17) |
| Pln(1)-Zr-Pln(2) | 128.61(15) | C(10)-Zr-C(6) | 32.28(17) |

| | | | |
|-----------------|------------|---------------------|----------|
| C(5)-Zr-C(6) | 149.71(18) | C(8)-C(7)-H(7) | 125.5 |
| C(2)-Zr-C(6) | 152.46(19) | C(6)-C(7)-H(7) | 125.5 |
| C(11)-Zr-C(1) | 87.25(18) | Zr-C(7)-H(7) | 117.9 |
| Cl-Zr-C(1) | 88.33(13) | C(7)-C(8)-C(9) | 107.9(5) |
| C(8)-Zr-C(1) | 133.44(18) | C(7)-C(8)-Zr | 74.5(3) |
| C(9)-Zr-C(1) | 132.01(17) | C(9)-C(8)-Zr | 73.7(3) |
| C(7)-Zr-C(1) | 152.40(18) | C(7)-C(8)-H(8) | 126.1 |
| C(4)-Zr-C(1) | 53.32(17) | C(9)-C(8)-H(8) | 126.1 |
| C(3)-Zr-C(1) | 53.23(18) | Zr-C(8)-H(8) | 117.7 |
| C(10)-Zr-C(1) | 149.5(2) | C(10)-C(9)-C(8) | 106.9(5) |
| C(5)-Zr-C(1) | 31.74(19) | C(10)-C(9)-Zr | 74.4(3) |
| C(2)-Zr-C(1) | 32.29(18) | C(8)-C(9)-Zr | 73.0(3) |
| C(6)-Zr-C(1) | 173.06(17) | C(10)-C(9)-H(9) | 126.5 |
| C(5)-C(1)-C(2) | 107.9(5) | C(8)-C(9)-H(9) | 126.5 |
| C(5)-C(1)-Zr | 73.7(3) | Zr-C(9)-H(9) | 118.1 |
| C(2)-C(1)-Zr | 73.5(3) | C(6)-C(10)-C(9) | 108.3(5) |
| C(5)-C(1)-H(1) | 126.1 | C(6)-C(10)-Zr | 74.6(3) |
| C(2)-C(1)-H(1) | 126.1 | C(9)-C(10)-Zr | 73.1(3) |
| Zr-C(1)-H(1) | 118.7 | C(6)-C(10)-H(10) | 125.9 |
| C(3)-C(2)-C(1) | 107.1(5) | C(9)-C(10)-H(10) | 125.9 |
| C(3)-C(2)-Zr | 73.0(3) | Zr-C(10)-H(10) | 118.3 |
| C(1)-C(2)-Zr | 74.2(3) | C(15)-C(11)-C(12) | 103.8(4) |
| C(3)-C(2)-H(2) | 126.4 | C(15)-C(11)-Zr | 117.3(3) |
| C(1)-C(2)-H(2) | 126.4 | C(12)-C(11)-Zr | 123.0(3) |
| Zr-C(2)-H(2) | 118.3 | C(15)-C(11)-H(11) | 103.4 |
| C(4)-C(3)-C(2) | 108.7(5) | C(12)-C(11)-H(11) | 103.4 |
| C(4)-C(3)-Zr | 73.7(3) | Zr-C(11)-H(11) | 103.4 |
| C(2)-C(3)-Zr | 74.7(3) | C(13)-C(12)-C(11) | 106.0(4) |
| C(4)-C(3)-H(3) | 125.7 | C(13)-C(12)-H(12A) | 110.5 |
| C(2)-C(3)-H(3) | 125.7 | C(11)-C(12)-H(12A) | 110.5 |
| Zr-C(3)-H(3) | 117.9 | C(13)-C(12)-H(12B) | 110.5 |
| C(5)-C(4)-C(3) | 107.4(5) | C(11)-C(12)-H(12B) | 110.5 |
| C(5)-C(4)-Zr | 74.8(3) | H(12A)-C(12)-H(12B) | 108.7 |
| C(3)-C(4)-Zr | 74.1(3) | C(14)-C(13)-C(12) | 104.8(4) |
| C(5)-C(4)-H(4) | 126.3 | C(14)-C(13)-H(13A) | 110.8 |
| C(3)-C(4)-H(4) | 126.3 | C(12)-C(13)-H(13A) | 110.8 |
| Zr-C(4)-H(4) | 116.9 | C(14)-C(13)-H(13B) | 110.8 |
| C(1)-C(5)-C(4) | 108.9(5) | C(12)-C(13)-H(13B) | 110.8 |
| C(1)-C(5)-Zr | 74.6(3) | H(13A)-C(13)-H(13B) | 108.9 |
| C(4)-C(5)-Zr | 72.9(3) | C(15)-C(14)-C(13) | 102.9(4) |
| C(1)-C(5)-H(5) | 125.6 | C(15)-C(14)-H(14B) | 111.2 |
| C(4)-C(5)-H(5) | 125.6 | C(13)-C(14)-H(14B) | 111.2 |
| Zr-C(5)-H(5) | 118.7 | C(15)-C(14)-H(14C) | 111.2 |
| C(7)-C(6)-C(10) | 108.0(5) | C(13)-C(14)-H(14C) | 111.2 |
| C(7)-C(6)-Zr | 72.8(3) | H(14B)-C(14)-H(14C) | 109.1 |
| C(10)-C(6)-Zr | 73.1(3) | C(14)-C(15)-C(11) | 104.5(4) |
| C(7)-C(6)-H(6) | 126.0 | C(14)-C(15)-H(15C) | 110.9 |
| C(10)-C(6)-H(6) | 126.0 | C(11)-C(15)-H(15C) | 110.9 |
| Zr-C(6)-H(6) | 119.9 | C(14)-C(15)-H(15A) | 110.9 |
| C(8)-C(7)-C(6) | 109.0(5) | C(11)-C(15)-H(15A) | 110.9 |
| C(8)-C(7)-Zr | 73.4(3) | H(15C)-C(15)-H(15A) | 108.9 |
| C(6)-C(7)-Zr | 75.1(3) | | |

Cent(1) is the centroid formed by C(1), C(2), C(3), C(4) and C(5).

Cent(2) is the centroid formed by C(6), C(7), C(8), C(9) and C(10).

Pln(1) is the plane formed by C(1), C(2), C(3), C(4) and C(5).

Pln(2) is the plane formed by C(6), C(7), C(8), C(9) and C(10).

Table 5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^4$) for \mathcal{S} The
 anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots$
 $+ 2 h k a^* b^* U^{12}]$

| | U^{11} | U^{22} | U^{33} | U^{23} | U^{13} | U^{12} |
|-------|----------|----------|----------|----------|----------|----------|
| Zr | 93(2) | 87(2) | 84(2) | 5(2) | -3(2) | 2(2) |
| Cl | 209(6) | 191(6) | 99(5) | 15(5) | 42(5) | -8(6) |
| C(1) | 190(30) | 70(20) | 270(30) | 20(20) | 90(30) | 50(20) |
| C(2) | 160(30) | 100(30) | 210(30) | -20(20) | -20(30) | 60(20) |
| C(3) | 120(30) | 90(30) | 280(30) | 20(20) | 80(30) | 30(20) |
| C(4) | 160(30) | 100(20) | 410(40) | -70(30) | -130(30) | 90(20) |
| C(5) | 300(40) | 130(30) | 150(30) | 20(20) | -40(30) | 130(30) |
| C(6) | 170(30) | 60(20) | 160(30) | -10(20) | 20(30) | 2(19) |
| C(7) | 200(30) | 70(20) | 130(30) | 19(19) | -40(30) | -50(30) |
| C(8) | 170(30) | 130(30) | 170(30) | 10(20) | 60(30) | -40(20) |
| C(9) | 100(30) | 110(20) | 290(30) | 0(30) | -30(30) | -30(20) |
| C(10) | 240(40) | 100(20) | 170(30) | -10(20) | -70(30) | -50(30) |
| C(11) | 90(20) | 140(30) | 120(20) | 0(20) | -10(20) | -30(20) |
| C(12) | 160(30) | 120(30) | 120(20) | 10(20) | -20(20) | 20(20) |
| C(13) | 160(30) | 170(30) | 200(30) | 0(20) | 0(20) | -10(20) |
| C(14) | 100(20) | 110(20) | 220(30) | 30(20) | -10(20) | 0(20) |
| C(15) | 130(30) | 150(30) | 160(30) | -10(20) | 10(20) | 0(20) |

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for β

| | x | y | z | U_{iso} |
|--------|-------|-------|------|------------------|
| H(1) | 1265 | 7239 | 6760 | 21 |
| H(2) | 1852 | 7824 | 4972 | 18 |
| H(3) | 4304 | 8893 | 4999 | 20 |
| H(4) | 5201 | 8998 | 6780 | 27 |
| H(5) | 3342 | 7945 | 7853 | 23 |
| H(6) | 648 | 12314 | 6811 | 15 |
| H(7) | 1209 | 11811 | 5028 | 16 |
| H(8) | 3846 | 11122 | 4918 | 19 |
| H(9) | 4955 | 11159 | 6681 | 20 |
| H(10) | 2937 | 11880 | 7845 | 20 |
| H(11) | -494 | 8876 | 5691 | 14 |
| H(12A) | 839 | 10003 | 4082 | 16 |
| H(12B) | -78 | 8834 | 4082 | 16 |
| H(13A) | -1231 | 11064 | 3772 | 21 |
| H(13B) | -1940 | 9887 | 3396 | 21 |
| H(14B) | -3268 | 10898 | 4832 | 17 |
| H(14C) | -3074 | 9542 | 4928 | 17 |
| H(15C) | -1956 | 10218 | 6383 | 18 |
| H(15A) | -1163 | 11245 | 5802 | 18 |