Crystal structure of 1,1′,2,2′,4,4′-hexaisopropyl-magnesocene

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The title compound, 3Cp2Mg or [Mg(C14H23)2], was synthesized from the corresponding triisopropylcyclopentadiene by treatment with n-butyl-sec-butylmagnesium. The structural characterization by single-crystal X-ray diffraction revealed that the compound crystallizes in the triclinic space group P1 with half a molecule per asymmetric unit and a staggered arrangement of the cyclopentadienide ligands.

1. Chemical context

Magnesocene (Cp2Mg) was initially reported by Wilkinson and Fischer and co-workers in 1954, just a few years after the discovery of ferrocene (Wilkinson & Cotton, 1954; Fischer & Hafner, 1954). Although magnesocene exhibits distinctively different chemical properties, it is isostructural to ferrocene and marked the beginning of main-group metallocene chemistry. One of the key differences in reactivity between alkaline-earth metallocenes and ferrocenes is that the central atoms of the former exhibit Lewis acidic character. Therefore, many crystal structures of magnesocenes are actually of donor complexes, such as magnesocene mono- and bis(tetrahydrofuran) adduct, Cp2Mg(thf) and Cp2Mg(thf)2 (Lehmkuhl et al., 1986; Jaenschke et al., 2003; Kim et al., 2007). Nevertheless, solvent-free crystal structures are also known, especially in case of highly substituted magnesocenes (Morley et al., 1987; Gardiner et al., 1991; Weber et al., 2002; Vollet et al., 2003; Deacon et al., 2015; Müller et al., 2021). Hanusa and coworkers had reported the synthesis of 1,1′,2,2′,4,4′-hexaisopropylmagnesocene, 3Cp2Mg, -calcocene, 3Cp2Ca, -strontocene, 3Cp2Sr, and -barocene, 3Cp2Ba (the triisopropylcyclopentadienide ligand is commonly abbreviated as ‘3Cp’), via treatment of potassium 1,2,4-triisopropylcyclopentadienide, 3CpK, with the corresponding metal(II) bromide or iodide and described the magnesocene to be oily or waxy in composition (Burkey et al., 1993, 1994). Thus, no crystal structure was obtained of the title compound. We found that the title compound may also be obtained through treatment of an isomeric mixture of triisopropylcyclopentadiene with n-butyl-sec-butylmagnesium in hexane.
2. Structural commentary

The title compound crystallizes in the triclinic space group $P\overline{1}$ with half a molecule per asymmetric unit, due to an inversion center at the magnesium atom position (Fig. 1), resulting in $C_2h$ symmetry for the molecule. Accordingly, the C₅ rings adopt a staggered arrangement with the single isopropyl group at the C₄ position facing the two isopropyl groups at the C₁ and C₂ positions and are perfectly coplanar to each other (Fig. 2). The C–C bond lengths within the C₅ ring are almost equal [C₁–C₂: 1.4237 (18) Å; C₂–C₃: 1.4268 (17) Å; C₃–C₄: 1.4172 (19) Å; C₄–C₅: 1.4220 (18) Å; C₅–C₁: 1.4277 (18) Å] implying a high degree of 6/25 electron aromaticity, and the Mg–C₅ centroid distance is 1.9852 (1) Å, which is within the normal range [e.g.: Cp₂Mg: 1.9897 (5) Å] for magnesocenes (Bünker & Weiss, 1975).

3. Supramolecular features

The molecules of the title compound are well separated from each other in the crystal structure, with one magnesocene molecule per unit cell. Each molecule has eight neighboring molecules, forming a distorted hexagonal bipyramidal coordination geometry (Fig. 3a and 3b), with distances of $d_{\text{min}}$ (Mg₁–Mg₁') = 8.7025 (4) Å, $d_{\text{max}}$ (Mg₁–Mg₁''') = 9.3031 (3) Å, and $d_{\text{axial}}$ (Mg₁–Mg₁'') = 9.2033 (4) Å [symmetry codes: (i) 1 + x, y, z; (ii) 1 + x, -1 + y, z; (iii) 1 + x, -1 + y, z; (iv) x, y, 1 + z]. The angles between the equatorial Mg atoms, the central magnesium atom and the axial magnesium atom are between $\theta_{\text{min}} = 90.68^\circ$ (Mg₁''–Mg₁–Mg₁''') and $\theta_{\text{max}} = 99.17^\circ$ (Mg₁''–Mg₁–Mg₁''').

Each Cp₂Mg moiety has eight neighboring molecules within the bc and ac planes (Fig. 4a and 4b), but only six neighboring molecules within the ab plane, forming an almost hexagonal layer ($\gamma = 63.00^\circ$), but with the layers being congruent to each other (Fig. 4c).

4. Database survey

A search in the Cambridge Structural Database (CSD, Version 5.42, update of September 2021; Groom et al., 2016) showed that 14 crystal structures of magnesocenes of the type (C₅R₅)₂Mg had previously been reported. In this search, any type of donor complexes of magnesocenes of the form (C₅R₅)₂Mg–Dₓ are not counted. The Mg–C₅ centroid bonding distances in these structures lie between 1.9562 (1) and 2.0628 (11) Å and the dihedral angles between the C₅ planes are between 0° (co-planar geometry) and 17.892°. Thus, the bond distances and angles in the title compound are within normal ranges of known magnesocenes.
5. Synthesis and crystallization

Hanusa and coworkers had previously reported that 1,1’,2’,2’,4,4’-hexaisopropylnorbornene, \( ^3\text{Cp}_2\text{Mg} \), could be obtained by the reaction of potassium 1,2,4-trisopropylcyclopentadienide with magnesium(II) bromide. However, in this work, we utilized dibutylmagnesium as a strong base to deprotonate the triisopropylcyclopentadiene (Fig. 5).

To a solution of 4.00 g (20.8 mmol) of an isomeric mixture of triisopropylcyclopentadiene in 100 mL of hexane were added 15.0 mL of a 0.7 M solution of \( n\)-butyl-sec-butylmagnesium in hexane (10.5 mmol). The light-yellow reaction solution was stirred at 333 K overnight. Subsequently, all volatiles were removed in vacuo and a yellow oil was obtained, from which the title compound crystallized over the course of one day at ambient temperature. The crystallized material was washed with small portions of hexane and dried in vacuo to obtain the title compound as a pure, colorless, crystalline solid in 43% yield (1.83 g; 4.50 mmol).

In addition to a structural characterization by single-crystal X-ray diffraction, the title compound was also characterized by \( ^1\text{H} \) and \( ^{13}\text{C} \) NMR spectroscopy: \( ^1\text{H} \) NMR (400 MHz, C\(_6\)D\(_6\), 295 K): \( \delta \) (in ppm) = 1.07 \([d, J = 7\text{Hz}, 12\text{H}, \text{CH}(\text{CH}_3)_2]\), 1.28 \([d, J = 7\text{Hz}, 12\text{H}, \text{CH}(\text{CH}_3)_2]\), 2.82–2.92 \([m, 6\text{H}, \text{CH}(\text{CH}_3)_2]\), 5.77 \([s, 4\text{H}, \text{Cp-H}]\); \( ^1\text{H} \) NMR (400 MHz, DMSO-D\(_6\), 294 K): \( \delta \) (in ppm) = 1.06 \([d, J = 7\text{Hz}, 36\text{H}, \text{CH}(\text{CH}_3)_2]\), 2.68 \([\text{sep}, J = 7\text{Hz}, 2\text{H}, \text{CH}(\text{CH}_3)_2]\), 2.76 \([\text{sep}, J = 7\text{Hz}, 2\text{H}, \text{CH}(\text{CH}_3)_2]\), 4.94 \([s, 4\text{H}, \text{Cp-H}]\); \( ^{13}\text{C}[^1\text{H}] \) NMR (101 MHz, C\(_6\)D\(_6\), 295 K): \( \delta \) (in ppm) = 24.0 \((\text{Pr})\), 24.4 \((\text{Pr})\), 26.4 \((\text{Pr})\), 26.6 \((\text{Pr})\), 28.7 \((\text{Pr})\), 98.7 \((\text{Cp})\), 125.3 \((\text{Cp})\), 128.6 \((\text{Cp})\); \( ^{13}\text{C}[^3\text{H}] \) NMR (101 MHz, DMSO-D\(_6\), 294 K): \( \delta \) (in ppm) = 25.9 \((\text{Pr})\), 26.8 \((\text{Pr})\), 27.0 \((\text{Pr})\), 29.1 \((\text{Pr})\), 94.6 \((\text{Cp})\), 119.4 \((\text{Cp})\), 120.9 \((\text{Cp})\).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All non-H atoms were placed in the electron density maps and refined anisotropically. C-bound H atoms were placed in positions of optimized geometry and treated as riding atoms: C—H = 1.00 Å (CH), 0.98 Å (CH\(_3\)), and with \( U_{iso}(\text{H}) = kU_{eq}(\text{C}) \), where \( k = 1.2 \) for CH and 1.5 for CH\(_3\).

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supporting information

Crystal structure of 1,1',2,2',4,4'-hexaisopropylmagnesocene

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Computing details

Data collection: APEX3 (Bruker, 2019); cell refinement: SAINT (Bruker, 2019); data reduction: SAINT (Bruker, 2019); program(s) used to solve structure: SHELXT2018/2 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/3 (Sheldrick, 2015b), shelXle (Hübschle et al., 2011); software used to prepare material for publication: publCIF (Westrip, 2010).

1,1',2,2',4,4'-Hexaisopropylmagnesocene

Crystal data

\[\text{Mg(C}_14\text{H}_{23})_2\]  
Mr = 406.96  
Triclinic, P\(\overline{1}\)  
\(a = 8.7025\) (4) Å  
\(b = 9.0903\) (4) Å  
\(c = 9.2033\) (4) Å  
\(\alpha = 80.829\) (2)°  
\(\beta = 81.151\) (2)°  
\(\gamma = 63.004\) (1)°  
\(V = 637.68\) (5) Å\(^3\)  
Z = 1  
\(F(000) = 226\)  
\(D_x = 1.060\) Mg m\(^{-3}\)  
Mo Ka radiation, \(\lambda = 0.71073\) Å  
Cell parameters from 7985 reflections  
\(\theta = 2.5–27.1°\)  
\(\mu = 0.08\) mm\(^{-1}\)  
\(T = 133\) K  
Plate, yellow  
0.27 × 0.20 × 0.07 mm

Data collection

Bruker D8 Venture Photon II diffractometer  
Radiation source: INCOATEC IÅµS microfocus sealed tube  
\(\varphi\) and \(\omega\) scans  
Absorption correction: multi-scan  
(SADABS; Krause et al., 2015)  
\(T_{\text{min}} = 0.712, T_{\text{max}} = 0.746\)  
24343 measured reflections  
2808 independent reflections  
2339 reflections with \(I > 2\sigma(I)\)  
\(R_{\text{int}} = 0.046\)  
\(\theta_{\text{max}} = 27.1°, \theta_{\text{min}} = 2.3°\)  
\(h = -11\rightarrow 11\)  
\(k = -11\rightarrow 11\)  
\(l = -11\rightarrow 11\)

Refinement

Refinement on \(F^2\)  
Least-squares matrix: full  
\(R[F^2 > 2\sigma(F^2)] = 0.041\)  
\(wR(F^2) = 0.100\)  
\(S = 1.06\)  
2808 reflections  
138 parameters  
0 restraints  
Primary atom site location: structure-invariant direct methods  
Secondary atom site location: difference Fourier map  
Hydrogen site location: inferred from neighbouring sites  
H-atom parameters constrained  
\(w = 1/[\sigma(F_c^2) + (0.0357P)^2 + 0.2575P]\)  
where \(P = (F_c^2 + 2F_s^2)/3\)  
\(\Delta/\sigma)_{\text{max}} < 0.001\)  
\(\Delta\rho_{\text{max}} = 0.23\) e Å\(^{-3}\)  
\(\Delta\rho_{\text{min}} = -0.20\) e Å\(^{-3}\)
**Special details**

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

| Atom | x       | y       | z       | Uiso* / Ueq |
|------|---------|---------|---------|-------------|
| Mg1  | 0.50000 | 0.50000 | 0.50000 | 0.02198 (16) |
| C1   | 0.58902 (16) | 0.29370 (15) | 0.34104 (14) | 0.0220 (3) |
| C2   | 0.68748 (15) | 0.38509 (15) | 0.29476 (14) | 0.0222 (3) |
| C3   | 0.78805 (16) | 0.36201 (16) | 0.41245 (14) | 0.0230 (3) |
| H3   | 0.876899 | 0.404101 | 0.408093 | 0.028* |
| C4   | 0.75424 (15) | 0.25725 (15) | 0.53102 (14) | 0.0223 (3) |
| C5   | 0.63013 (16) | 0.21621 (15) | 0.48667 (14) | 0.0232 (3) |
| H5   | 0.587559 | 0.136717 | 0.544119 | 0.028* |
| C6   | 0.47318 (17) | 0.27043 (16) | 0.24895 (15) | 0.0257 (3) |
| H6   | 0.444736 | 0.359348 | 0.164188 | 0.031* |
| C7   | 0.30301 (19) | 0.2858 (2) | 0.33539 (19) | 0.0371 (4) |
| H7A  | 0.233030 | 0.269800 | 0.271125 | 0.056* |
| H7B  | 0.327426 | 0.201156 | 0.420434 | 0.056* |
| H7C  | 0.239326 | 0.396273 | 0.370110 | 0.056* |
| C8   | 0.5672 (2) | 0.10251 (19) | 0.18531 (18) | 0.0368 (4) |
| H8A  | 0.491531 | 0.091596 | 0.123487 | 0.055* |
| H8B  | 0.597174 | 0.012997 | 0.266371 | 0.055* |
| H8C  | 0.673219 | 0.095503 | 0.125346 | 0.055* |
| C9   | 0.69613 (17) | 0.47897 (17) | 0.14521 (15) | 0.0261 (3) |
| H9   | 0.585123 | 0.513065 | 0.101394 | 0.031* |
| C10  | 0.7151 (2) | 0.6358 (2) | 0.15456 (18) | 0.0422 (4) |
| H10A | 0.719212 | 0.692389 | 0.055076 | 0.063* |
| H10B | 0.822300 | 0.606089 | 0.198629 | 0.063* |
| H10C | 0.615986 | 0.709829 | 0.215971 | 0.063* |
| C11  | 0.8434 (2) | 0.3681 (2) | 0.04173 (17) | 0.0401 (4) |
| H11A | 0.843621 | 0.430001 | −0.055715 | 0.060* |
| H11B | 0.827146 | 0.270336 | 0.032239 | 0.060* |
| H11C | 0.954060 | 0.332769 | 0.082140 | 0.060* |
| C12  | 0.82800 (17) | 0.20815 (17) | 0.68044 (15) | 0.0274 (3) |
| H12  | 0.750729 | 0.296459 | 0.745989 | 0.033* |
| C13  | 1.00801 (19) | 0.1994 (2) | 0.66748 (18) | 0.0408 (4) |
| H13A | 1.087252 | 0.112649 | 0.604724 | 0.061* |
| H13B | 1.049205 | 0.173474 | 0.765945 | 0.061* |
| H13C | 1.003708 | 0.306602 | 0.623400 | 0.061* |
| C14  | 0.8308 (3) | 0.0458 (2) | 0.75386 (19) | 0.0463 (4) |
| H14A | 0.712755 | 0.055338 | 0.767566 | 0.069* |
| H14B | 0.876845 | 0.019984 | 0.850239 | 0.069* |
| H14C | 0.904440 | −0.043197 | 0.691324 | 0.069* |
### Atomic displacement parameters (Å²)

|        | \( U^{11} \)   | \( U^{22} \)   | \( U^{33} \)   | \( U^{12} \)   | \( U^{13} \)   | \( U^{23} \)   |
|--------|----------------|----------------|----------------|----------------|----------------|----------------|
| Mg1    | 0.0202 (3)     | 0.0207 (3)     | 0.0236 (3)     | −0.0070 (2)    | 0.0002 (2)     | −0.0067 (2)    |
| C1     | 0.0213 (6)     | 0.0189 (6)     | 0.0243 (6)     | −0.0064 (5)    | −0.0012 (5)    | −0.0066 (5)    |
| C2     | 0.0195 (6)     | 0.0222 (6)     | 0.0227 (6)     | −0.0066 (5)    | 0.0004 (5)     | −0.0063 (5)    |
| C3     | 0.0183 (6)     | 0.0259 (6)     | 0.0246 (6)     | −0.0088 (5)    | −0.0004 (5)    | −0.0064 (5)    |
| C4     | 0.0191 (6)     | 0.0209 (6)     | 0.0238 (6)     | −0.0049 (5)    | −0.0011 (5)    | −0.0065 (5)    |
| C5     | 0.0247 (6)     | 0.0196 (6)     | 0.0247 (6)     | −0.0089 (5)    | −0.0015 (5)    | −0.0040 (5)    |
| C6     | 0.0281 (7)     | 0.0234 (6)     | 0.0278 (7)     | −0.0116 (5)    | −0.0063 (5)    | −0.0040 (5)    |
| C7     | 0.0287 (7)     | 0.0408 (8)     | 0.0478 (9)     | −0.0168 (6)    | −0.0016 (6)    | −0.0174 (7)    |
| C8     | 0.0358 (8)     | 0.0375 (8)     | 0.0415 (9)     | −0.0151 (7)    | −0.0044 (7)    | −0.0184 (7)    |
| C9     | 0.0246 (6)     | 0.0300 (7)     | 0.0236 (7)     | −0.0123 (5)    | −0.0010 (5)    | −0.0027 (5)    |
| C10    | 0.0609 (11)    | 0.0354 (8)     | 0.0335 (8)     | −0.0274 (8)    | 0.0068 (7)     | −0.0033 (7)    |
| C11    | 0.0467 (9)     | 0.0395 (8)     | 0.0274 (8)     | −0.0156 (7)    | 0.0090 (7)     | −0.0075 (6)    |
| C12    | 0.0268 (7)     | 0.0270 (6)     | 0.0246 (7)     | −0.0071 (5)    | −0.0034 (5)    | −0.0059 (5)    |
| C13    | 0.0298 (8)     | 0.0566 (10)    | 0.0338 (8)     | −0.0146 (7)    | −0.0090 (6)    | −0.0067 (7)    |
| C14    | 0.0652 (11)    | 0.0427 (9)     | 0.0356 (9)     | −0.0270 (9)    | −0.0204 (8)    | 0.0085 (7)     |

### Geometric parameters (Å, °)

| Bond                  | Distance (Å)   | Angle (°)   |
|-----------------------|----------------|-------------|
| Mg1—C3i               | 2.3136 (12)    | C7—H7B      | 0.9800      |
| Mg1—C3                | 2.3136 (12)    | C7—H7C      | 0.9800      |
| Mg1—C5                | 2.3148 (12)    | C8—H8A      | 0.9800      |
| Mg1—C5i               | 2.3148 (12)    | C8—H8B      | 0.9800      |
| Mg1—C4                | 2.3253 (12)    | C8—H8C      | 0.9800      |
| Mg1—C4i               | 2.3253 (12)    | C9—C11      | 1.5239 (19) |
| Mg1—C2i               | 2.3355 (12)    | C9—C10      | 1.525 (2)   |
| Mg1—C2                | 2.3355 (12)    | C9—H9       | 1.0000      |
| Mg1—C1i               | 2.3375 (12)    | C10—H10A    | 0.9800      |
| Mg1—C1                | 2.3376 (12)    | C10—H10B    | 0.9800      |
| C1—C2                | 1.4237 (18)    | C10—H10C    | 0.9800      |
| C1—C5                | 1.4277 (18)    | C11—H11A    | 0.9800      |
| C1—C6                | 1.5143 (17)    | C11—H11B    | 0.9800      |
| C2—C3                | 1.4268 (17)    | C11—H11C    | 0.9800      |
| C2—C9                | 1.5101 (18)    | C12—C14     | 1.514 (2)   |
| C3—C4                | 1.4172 (19)    | C12—C13     | 1.519 (2)   |
| C3—H3                | 1.0000         | C12—H12     | 1.0000      |
| C4—C5                | 1.4220 (18)    | C13—H13A    | 0.9800      |
| C4—C12               | 1.5226 (18)    | C13—H13B    | 0.9800      |
| C5—H5                | 1.0000         | C13—H13C    | 0.9800      |
| C6—C7                | 1.527 (2)      | C14—H14A    | 0.9800      |
| C6—C8                | 1.5317 (18)    | C14—H14B    | 0.9800      |
| C6—H6                | 1.0000         | C14—H14C    | 0.9800      |
| C7—H7A               | 0.9800         |              |             |
| C3i—Mg1—C3            | 180.0          | C5—C4—C12   | 126.88 (12) |
| C3i—Mg1—C5            | 121.04 (5)     | C3—C4—Mg1   | 71.76 (7)   |
| Bond          | Distance (Å) | Bond          | Distance (Å) |
|---------------|--------------|---------------|--------------|
| C3—Mg1—C5    | 58.96 (5)    | C5—C4—Mg1    | 71.75 (7)    |
| C3—Mg1—C5i   | 58.96 (5)    | C12—C4—Mg1   | 118.67 (8)   |
| C3—Mg1—C5i   | 121.04 (5)   | C4—C5—C1     | 109.12 (11)  |
| C5—Mg1—C5    | 120.0        | C4—C5—H5     | 72.56 (7)    |
| C3—Mg1—C4    | 144.42 (5)   | C1—C5—Mg1    | 73.00 (7)    |
| C3—Mg1—C4    | 35.58 (5)    | C4—C5—H5     | 125.3        |
| C5—Mg1—C4    | 35.69 (4)    | C1—C5—H5     | 125.3        |
| C5—Mg1—C4    | 144.31 (4)   | Mg1—C5—H5    | 125.3        |
| C3i—Mg1—C5i  | 58.96 (5)    | C1—C6—C7     | 112.68 (11)  |
| C3—Mg1—C4i   | 144.42 (5)   | C1—C6—C8     | 110.81 (11)  |
| C5—Mg1—C4i   | 144.31 (4)   | C7—C6—C8     | 109.65 (12)  |
| C5—Mg1—C4i   | 35.69 (4)    | C1—C6—H6     | 107.8        |
| C4—Mg1—C4i   | 180.0        | C7—C6—H6     | 107.8        |
| C3—Mg1—C2i   | 35.74 (4)    | C8—C6—H6     | 107.8        |
| C3—Mg1—C2i   | 144.26 (4)   | C6—C7—H7A    | 109.5        |
| C5—Mg1—C2i   | 120.74 (5)   | C6—C7—H7B    | 109.5        |
| C5—Mg1—C2i   | 59.26 (5)    | H7A—C7—H7B   | 109.5        |
| C4—Mg1—C2i   | 120.27 (4)   | C6—C7—H7C    | 109.5        |
| C4—Mg1—C2i   | 59.73 (4)    | H7A—C7—H7C   | 109.5        |
| C3i—Mg1—C2i  | 144.26 (4)   | C6—C8—H8A    | 109.5        |
| C3i—Mg1—C2i  | 35.74 (4)    | C6—C8—H8B    | 109.5        |
| C5—Mg1—C2i   | 59.26 (5)    | C6—C8—H8B    | 109.5        |
| C5—Mg1—C2i   | 120.74 (5)   | H8A—C8—H8B   | 109.5        |
| C4—Mg1—C2i   | 59.73 (4)    | C6—C8—H8C    | 109.5        |
| C4—Mg1—C2i   | 120.27 (4)   | H8A—C8—H8C   | 109.5        |
| C2—Mg1—C2i   | 180.0        | H8B—C8—H8C   | 109.5        |
| C3—Mg1—C1i   | 120.83 (4)   | C2—C9—C11    | 110.83 (11)  |
| C3—Mg1—C1i   | 144.26 (4)   | C2—C9—C10    | 112.69 (11)  |
| C5—Mg1—C1i   | 59.17 (4)    | C11—C9—C10   | 109.89 (12)  |
| C5—Mg1—C1i   | 120.28 (4)   | C2—C9—H9     | 107.7        |
| C4—Mg1—C1i   | 59.72 (4)    | C11—C9—H9    | 107.7        |
| C4—Mg1—C1i   | 35.48 (4)    | C10—C9—H9    | 107.7        |
| C2—Mg1—C1i   | 144.26 (4)   | C9—C10—H10A  | 109.5        |
| C2—Mg1—C1i   | 144.52 (4)   | C9—C10—H10B  | 109.5        |
| C3—Mg1—C1    | 120.83 (4)   | H10A—C10—H10B| 109.5        |
| C3—Mg1—C1    | 59.17 (4)    | C9—C10—H10C  | 109.5        |
| C5—Mg1—C1    | 35.74 (4)    | H10A—C10—H10C| 109.5        |
| C5—Mg1—C1    | 144.26 (4)   | H10B—C10—H10C| 109.5        |
| C4—Mg1—C1    | 59.72 (4)    | C9—C11—H11A  | 109.5        |
| C4—Mg1—C1    | 120.28 (4)   | C9—C11—H11B  | 109.5        |
| C2—Mg1—C1    | 144.52 (4)   | H11A—C11—H11B| 109.5        |
| C2—Mg1—C1    | 35.48 (4)    | C9—C11—H11C  | 109.5        |
| C1i—Mg1—C1   | 180.0        | H11A—C11—H11C| 109.5        |
| C2—C1—C5     | 107.47 (11)  | H11B—C11—H11C| 109.5        |
| C2—C1—C6     | 126.57 (12)  | C14—C12—C13  | 110.23 (13)  |
| C5—C1—C6     | 125.78 (12)  | C14—C12—C4   | 112.14 (12)  |
| C2—C1—Mg1    | 72.18 (7)    | C13—C12—C4   | 111.48 (12)  |
| C5—C1—Mg1    | 71.26 (7)    | C14—C12—H12  | 107.6        |
| Bond          | Distance (Å) | Bond          | Distance (Å) | Bond          | Distance (Å) |
|--------------|--------------|--------------|--------------|--------------|--------------|
| C6—C1—Mg1   | 125.75 (8)   | C13—C12—H12 | 107.6        |              |              |
| C1—C2—C3    | 107.35 (11)  | C4—C12—H12  | 107.6        |              |              |
| C1—C2—C9    | 126.99 (11)  | C12—C13—H13A| 109.5        |              |              |
| C3—C2—C9    | 125.51 (12)  | C12—C13—H13B| 109.5        |              |              |
| C1—C2—Mg1   | 72.34 (7)    | H13A—C13—H13B| 109.5      |              |              |
| C3—C2—Mg1   | 71.29 (7)    | C12—C13—H13C| 109.5        |              |              |
| C9—C2—Mg1   | 125.19 (8)   | H13A—C13—H13C| 109.5      |              |              |
| C4—C3—C2    | 109.38 (11)  | H13B—C13—H13C| 109.5      |              |              |
| C4—C3—Mg1   | 72.66 (7)    | C12—C14—H14A| 109.5        |              |              |
| C2—C3—Mg1   | 72.97 (7)    | C12—C14—H14B| 109.5        |              |              |
| C4—C3—H3    | 125.2        | H14A—C14—H14B| 109.5      |              |              |
| C2—C3—H3    | 125.2        | C12—C14—H14C| 109.5        |              |              |
| Mg1—C3—H3   | 125.2        | H14A—C14—H14C| 109.5      |              |              |
| C3—C4—C5    | 106.69 (11)  | H14B—C14—H14C| 109.5      |              |              |
| C3—C4—C12   | 126.32 (12)  |              |              |              |              |

Symmetry code: (i) −x+1, −y+1, −z+1.