Characterization of 2,6-Trip$_2$H$_3$C$_6$GaCl$_2$, {2,6-Trip$_2$H$_3$C$_6$InCl(μ-Cl)}$_2$ and {2,6-Trip$_2$H$_3$C$_6$GaCl(μ-OH)}$_2$ (Trip = 2,4,6-triisopropylphenyl)

Brendan Twamley and Philip P. Power*

Department of Chemistry, University of California, Davis, One Shields Avenue, Davis, CA 95616 USA.
E-mail: pppower@ucdavis.edu

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The spectroscopy and structures of the title compounds show that the analogous species described previously were probably contaminated by OH$^-$ at the Cl$^-$ positions.

A recent paper in this journal reported the terphenyl gallium and indium dichlorides {2,6-Trip$_2$H$_3$C$_6$MCl$_2$}$_2$ (M = Ga or In; Trip = C$_6$H$_2$Pr$^i_3$-2,4,6) which were described as having halide bridged dimeric structures.[1] Unusually, the bridging metal chloride distances [Ga–μ-Cl 2.198(5) Å, In–μ-Cl 2.235(4) Å] were shorter than those to the terminal chlorides [Ga-Cl(terminal) 2.230(5) Å, In–Cl(terminal) 2.448(7) Å]. This differs from other halide bridged structures, including the related {2,6-Mes$_2$H$_3$C$_6$MCl$_2$}$_2$ (Mes = C$_6$H$_2$Me$_3$-2,4,6; M = Ga, In) dimers,[2] where the M–μ-Cl distances are longer than the terminal ones.[3] The M–μ-Cl bonds also differed by only 0.037 Å in spite of the different sizes of Ga and In.† Furthermore, the cell constants for these compounds resemble those reported for {2,6-Trip$_2$H$_3$C$_6$Al(Cl)$_{0.68}$(H)$_{0.32}$(μ-OH)}$_2$·2C$_6$H$_6$, which arose *via* contamination of the product (presumably by H$_2$O) during the synthesis of the halide {2,6-Trip$_2$H$_3$C$_6$AlCl$_2$}$_2$ from {2,6-Trip$_2$H$_3$C$_6$AlH$_2$}$_2$.[4] We hypothesized that the short 'Ga–Cl' and 'In–Cl' distances might be due to contamination of the μ-Cl positions by OH$^-$. We resolved to isolate the terphenyl metal dihalides under the rigorous exclusion of moisture, and to react these dihalide products with H$_2$O in order to synthesize the hydroxides deliberately.

Reaction of 2,6-Trip$_2$H$_3$C$_6$Li(OEt$_2$)[5] and GaCl$_3$ in Et$_2$O gave, initially, 2,6-Trip$_2$H$_3$C$_6$GaCl$_2$(OEt$_2$) **1a** as colorless crystals‡ which were characterized by spectroscopy. Its structure probably resembles that reported for 2,6-Mes$_2$H$_3$C$_6$AlCl$_2$(OEt$_2$).[4] Recrystallization of **1a** from hot hexane yielded 2,6-Trip$_2$H$_3$C$_6$GaCl$_2$ **1b**,‡ whose crystal structure shows that, unlike the earlier compound,[1] it is monomeric (Fig. 1).§ The Ga atom has distorted trigonal planar coordination with Ga–Cl 2.113(4) and 2.124(3) Å and Ga–C 1.930(8) Å. These are similar to those in the only other three-coordinate, monomeric aryl gallium dichloride, Mes*GaCl$_2$[6] (Mes* = C$_6$H$_2$Bu$^t_3$-2,4,6) which was characterized by two groups who reported the bond distances: Ga–C 1.935(4) Å (av.), Ga–Cl 2.157(1) Å (av.); Ga–C 1.953(13) Å, Ga–Cl 2.212(4) Å.

The reaction of 2,6-Trip$_2$H$_3$C$_6$Li(OEt$_2$)[5] with InCl$_3$ gave {2,6-Trip$_2$H$_3$C$_6$InCl$_2$}$_2$ **2** which was characterized by spectroscopy‡ and X-ray diffraction.§ There is ^{1}H NMR evidence that an Et$_2$O adduct of **2** (similar to **1a**) is formed, but dissociation of Et$_2$O is readily effected by recrystallization from hexane. It crystallizes as a centrosymmetric dimer (Fig. 2) whose structural details differ markedly from those previously reported.[1] Notably, the distance of 2.5239(7) Å is *ca.* 0.28 Å longer than the In–μ-Cl lengths 2.236(4) and 2.233(4) Å given earlier.[1] Also, the terminal In–Cl distance in **2**, 2.3341(12) Å, is *ca.* 0.11 Å shorter than the published In–Cl(terminal) distance, 2.448(7) Å. The In–C and In–Cl distances in **2** are in agreement with those in other halide bridged In compounds, *e.g.* {2,6-Mes$_2$H$_3$C$_6$InCl$_2$}$_2$,[2] In–μ-Cl 2.519(2), 2.514(2) Å, In–Cl(terminal) 2.344(3) Å; {In(CH$_2$CMe$_3$)(CH$_2$SiMe$_3$)Cl}$_2$,[3d] In–μ-Cl 2.659(3), 2.572(3) Å.

To show that the original structure of '{2,6-Trip$_2$H$_3$C$_6$GaCl$_2$}$_2$'[1] could have been contaminated with OH$^-$ at the bridging positions, H$_2$O was added to **1b** in THF.‡ Crystals of {2,6-Trip$_2$H$_3$C$_6$GaCl(μ-OH)}$_2$·2C$_6$H$_6$ **3** have cell parameters which resemble those of '{2,6-Trip$_2$H$_3$C$_6$GaCl$_2$}$_2$'[1] and {2,6-Trip$_2$H$_3$C$_6$Al(Cl)$_{0.68}$(H)$_{0.32}$(μ-OH)}$_2$·2C$_6$H$_6$.[4] It has a centrosymmetric OH$^-$ bridged structure with terminal Ga–Cl bonds (Fig. 3).§ The Ga–μ-O distances, 1.932(4) and 1.906(4) Å, are close to those in related OH$^-$ bridged compounds, *e.g.* {2,4,6-(CF$_3$)$_3$H$_2$C$_6$GaCl(μ-OH)}$_2$,[7] Ga-O 1.903(4), 1.895(4) Å; {Mes$_2$Ga(μ-OH)}$_2$·THF,[8] Ga–O 1.949(2) Å; {[((Me$_3$Si)$_2$CH)$_2$Ga(μ-OH)}$_2$,[9] Ga–O 1.963(4), 1.976(4) Å. The terminal Ga–Cl and Ga–C distances, 2.1465(19) and 1.949(5) Å, in **3** are similar to those in **1b** and related compounds.

The data for **1b**, **2** and **3** lead to the following conclusions: (a) 2,6-Trip$_2$H$_3$C$_6$GaCl$_2$ **1b** is a monomer owing to the large size of the C$_6$H$_3$Trip-2,6 ligand and the dimeric structure of the In compound **2** is due to the larger size of In which reduces steric crowding and permits association; (b) the dimeric structure of **3** results from contamination of the μ-Cl$^-$ ligands by OH$^-$, and since the originally reported short 'Ga–Cl' and 'In–Cl' distances lie about halfway between those expected for bridging

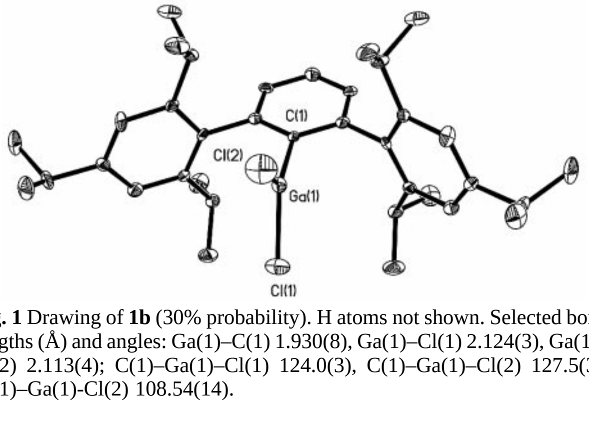

Fig. 1 Drawing of **1b** (30% probability). H atoms not shown. Selected bond lengths (Å) and angles: Ga(1)–C(1) 1.930(8), Ga(1)–Cl(1) 2.124(3), Ga(1)–Cl(2) 2.113(4); C(1)–Ga(1)–Cl(1) 124.0(3), C(1)–Ga(1)–Cl(2) 127.5(3), Cl(1)–Ga(1)-Cl(2) 108.54(14).

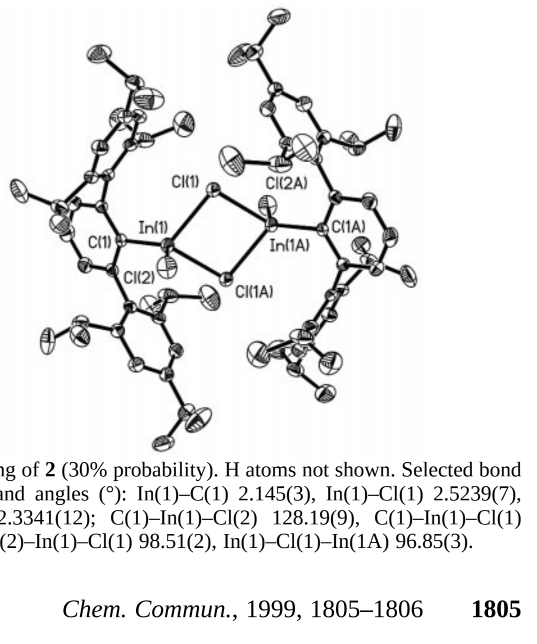

Fig. 2 Drawing of **2** (30% probability). H atoms not shown. Selected bond lengths (Å) and angles (°): In(1)–C(1) 2.145(3), In(1)–Cl(1) 2.5239(7), In(1)–Cl(2) 2.3341(12); C(1)–In(1)–Cl(2) 128.19(9), C(1)–In(1)–Cl(1) 119.01(6), Cl(2)–In(1)–Cl(1) 98.51(2), In(1)–Cl(1)–In(1A) 96.85(3).

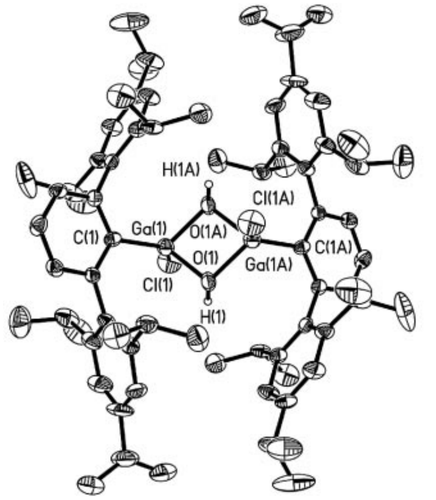

Fig. 3 Drawing of the structure of **3** (30% probability). H atoms not shown. Selected bond lengths (Å) and angles (°): Ga(1)–C(1) 1.949(5), Ga(1)–O(1) 1.932(4), Ga(1)–(O1A) 1.906(4), Ga(1)–Cl(1) 2.1465(19); C(1)–Ga(1)–Cl(1) 126.23(17), Ga(1)–O(1)–Ga(1A) 99.94(19), O(1A)–Ga(1)–Cl(1) 104.33(14).

M–OH and M–Cl (M = Ga, In) bonds, the contamination of the μ–Cl$^-$ positions by OH$^-$ ions is 50% which is sufficient to produce the apparent 'dimerization'; (c) the M–Cl distances in both **1b** and **2** are normal, and there is no shortening in the bridging In–Cl distance which is *ca*. 0.29 Å longer than the terminal In–Cl bond.

We note that the hydrolysis of related organogallium compounds to give Ga–OH products has been investigated in detail,[8,10] and the susceptibility of **1b** and **2** to hydrolysis agrees with these studies. The ability of OH$^-$ ligands to occupy Cl$^-$ sites bears some similarity to the bond stretch isomerism controversy where 'short' M–Cl (or 'long' M–O) bonds were shown to be a result of occupancy of the same site by different percentages of the O or Cl atoms rather than a new type of bonding isomerism.[11]

Notes and references

† The effective ionic radius of four coordinate Ga^{3+} is 0.47 Å and that of In^{3+} is 0.62 Å: R. D. Shannon, *Acta Crystallogr*., *Sect. A*, 1976, **32**, 751.

‡ *Experimental*: **1a**: under anaerobic and anhydrous conditions, 2,6-Trip$_2$-H$_3$C$_6$Li·OEt$_2$ (7.90 g, 14 mmol) in Et$_2$O (40 mL) at 0 °C was added dropwise (0.5 h) to freshly sublimed GaCl$_3$ (2.47 g, 14 mmol) in Et$_2$O (20 mL) at *ca*. −78 °C. The mixture was stirred at *ca*. −78 °C (3 h) before warming to *ca*. 20 °C. After stirring for 2 d, the solvent was removed and the residue was extracted with hexane (80 mL) and filtered (Celite). Concentration to incipient crystallization (*ca*. 40 mL) and cooling to *ca*. −25 °C yielded colorless crystals of **1a**, 7.60 g, 78%. Anal. calc. (found) for C$_{40}$H$_{59}$Cl$_2$GaO: C, 68.98 (68.53), H, 8.54 (8.91)%. ^{1}H NMR (300 MHz, C$_6$D$_6$) δ 0.82 [t, 6H, (C*H*$_3$CH$_2$)$_2$O], 1.15 [d, 12H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.6 Hz], 1.29 [d, 12H, *p*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 1.53 [d, 12H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.0 Hz], 2.88 [sept, 2H, *p*-C*H*(CH$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.6 Hz], 3.14 [q, 4H + 4H, (CH$_3$C*H*$_2$)$_2$O + *o*-C*H*(CH$_3$)$_2$, J_{HH} 6.9 Hz], 7.19–7.28 [m, 7H, *p*-C$_6$H$_3$, *m*-Trip (7.24, s), *m*-C$_6$H$_3$]; ^{13}C{^{1}H} NMR (75 MHz, C$_6$D$_6$) δ 14.3 [(*C*H$_3$CH$_2$)$_2$O], 23.1 [*p*-CH(*C*H$_3$)$_2$], 24.4 [*o*-CH(*C*H$_3$)$_2$], 26.2 [*o*-CH(*C*H$_3$)$_2$], 31.1 [*o*-*C*H(CH$_3$)$_2$], 34.8 [*p*-*C*H(CH$_3$)$_2$], 66.1 [(CH$_3$*C*H$_2$)$_2$O)], 121.0 (*m*-Trip), 128.2 (*p*-C$_6$H$_3$), 130.1 (*m*-C$_6$H$_3$), 138.8 (*ipso*-Trip), 147.4 (*p*-Trip), 147.8 (*o*-Trip), 148.8 (br, *o*-C$_6$H$_3$), the *ipso* C resonance was not observed. **1b**: **1a** (6.5 g, 0.93 mmol) in hexane (100 mL) was concentrated to *ca*. 50 mL. The precipitates were redissolved by heating and **1b** crystallized on standing at *ca*. 20 °C. Yield 5.0 g, 87%. Mp 230 °C sweats, 244 °C partial melt, 258–260 °C complete melt. Anal. calc. (found) for C$_{36}$H$_{49}$Cl$_2$Ga: C, 69.47 (68.90), H, 7.94 (8.11)%. IR $\nu_{\mathrm{Ga-Cl}}$ 400s, 330s br cm^{-1}; ^{1}H NMR (300 MHz, C$_6$D$_6$) δ 1.06 [d, 12H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 1.26 [d, 12H, *p*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 1.42 [d, 12H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 2.84 [sept, 2H, *p*-C*H*(CH$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 2.94 [sept, 4H, *o*-C*H*(CH$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 7.17–7.24 [m, 7H, *p*-C$_6$H$_3$, *m*-Trip (7.21, s), *m*-C$_6$H$_3$]; ^{13}C{^{1}H} NMR (75 MHz, C$_6$D$_6$) δ 22.5 [*p*-CH(*C*H$_3$)$_2$], 24.3 [*o*-CH(*C*H$_3$)$_2$], 26.3 [*o*-CH(*C*H$_3$)$_2$], 31.1 [*o*-*C*H(CH$_3$)$_2$], 34.8 [*p*-*C*H(CH$_3$)$_2$], 121.8 (*m*-Trip), 128.3 (*p*-C$_6$H$_3$), 129.1 (*m*-C$_6$H$_3$), 137.8 (*ipso*-Trip), 146.8 (*p*-Trip), 147.3 (*o*-Trip), 150.3 (*o*-C$_6$H$_3$), 171.6 (*ipso*-C$_6$H$_3$). **2**: A solution of 2,6-Trip$_2$H$_3$C$_6$Li·OEt$_2$ (3.95 g, 7.02 mmol) in Et$_2$O (40 mL) was added to a suspension of InCl$_3$ (1.55 g, 7.02 mmol) in Et$_2$O (20 mL) at *ca*. −78 °C. The mixture was stirred at ca. −78° C (1 h), before warming to *ca*. 20 °C, and stirred overnight. The solvent was removed and the residue was extracted with hexane (100 mL), and filtered (Celite). The pale yellow solution was concentrated to *ca*. 20 mL and heated to redissolve the precipitates. Standing at *ca*. 20 °C afforded colorless crystalline **2**, 3.74 g, 80%. Mp 160 °C opaque, 226 °C sweats, 236–238 °C melt. Anal. calc. (found) for C$_{72}$H$_{98}$Cl$_4$In$_2$: C, 64.78 (64.01), H, 7.39 (8.2)%. IR $\nu_{\mathrm{In-Cl}}$ 335s br cm^{-1}; ^{1}H NMR (300 MHz, C$_6$D$_6$) δ 1.05 [d, 24H, *p*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.6 Hz], 1.36 [d, 24H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 1.44 [d, 24H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 2.90 [m, 12H, *p*-C*H*(CH$_3$)$_2$ + *o*-C*H*(CH$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.6 Hz], 7.11 (t, 2H, *p*-C$_6$H$_3$, $^3J_{\mathrm{HH}}$ 7.5 Hz), 7.21 (s, 8H, *m*-Trip), 7.22 (d, 4H, *m*-C$_6$H$_3$, $^3J_{\mathrm{HH}}$ 13.2 Hz); ^{13}C{^{1}H} NMR (75 MHz, C$_6$D$_6$) δ 23.1 [*p*-CH(*C*H$_3$)$_2$], 24.4 [*o*-CH(*C*H$_3$)$_2$], 26.2 [*o*-CH(*C*H$_3$)$_2$], 31.0 [*o*-*C*H(CH$_3$)$_2$], 34.9 [*p*-*C*H(CH$_3$)$_2$], 121.8 (*m*-Trip), 128.9 (*p*-*C*$_6$H$_3$), 129.8 (*m*-*C*$_6$H$_3$), 138.5 (*ipso*-Trip), 147.1 (*p*-Trip), 148.1 (*o*-Trip), 149.8 (*o*-*C*$_6$H$_3$), *ipso* C resonance not observed. **3**: H$_2$O (33 μL, 1.85 mmol) was added to **1b** (1.15 g, 1.85 mmol) in THF (40 mL) at *ca*. 0 °C and stirred (2 d). The solvent was removed under reduced pressure and the residue was extracted with C$_6$H$_6$ (40 mL), filtered (Celite) and concentrated to *ca*. 10 mL. The solution was cooled to *ca*. 5 °C (2 weeks) to afford **3** as colorless crystals, 1.08 g, 90%. Anal. calc. (found) for C$_{72}$H$_{100}$Cl$_2$Ga$_2$O$_2$: C, 71.59 (72.31); H, 8.34 (8.62)%. Mp 215 °C sweats, 227–229 °C melt. IR ν_{OH} 3440br, 3200br, $\nu_{\mathrm{Ga-Cl}}$ 380s, 330s br cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$) δ 0.94 [d, 24H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 1.13 [d, 24H, *o*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.6 Hz], 1.17 [d, 24H, *p*-CH(C*H*$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.75 Hz], 2.58 [sept, 8H, *o*-C*H*(CH$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.75 Hz], 2.815 (sept, 4H, *p*-C*H*(CH$_3$)$_2$, $^3J_{\mathrm{HH}}$ 6.9 Hz], 4.00 (s, br 2H, OH), 6.97 (s, 8H, *m*-Trip), 7.10 (d, 4H, *m*-C$_6$H$_3$, $^3J_{\mathrm{HH}}$ 7.5 Hz), 7.31 (t, 2H, *p*-C$_6$H$_3$, $^3J_{\mathrm{HH}}$ 7.5 Hz); ^{13}C{^{1}H} NMR (75 MHz, CDCl$_3$) δ 22.6 [*p*-CH(*C*H$_3$)$_2$], 24.1 [*o*-CH(*C*H$_3$)$_2$], 25.7 [*o*-CH(*C*H$_3$)$_2$], 30.5 [*o*-*C*H(CH$_3$)$_2$], 34.4 [*p*-*C*H(CH$_3$)$_2$], 120.9 (*m*-Trip), 127.9 (*p*-C$_6$H$_3$), 128.9 (*m*-C$_6$H$_3$), 137.8 (*ipso*-Trip), 146.6 (*p*-Trip), 147.4 (*o*-Trip), 149.2 (*o*-*C*$_6$H$_3$), *ipso C* resonance not observed.

§ *Crystal data* at 130 K for **1b** and 299 K for **2** and **3**, with Mo-Kα (λ = 0.71073 Å) radiation: **1b**: C$_{36}$H$_{49}$Cl$_2$Ga, M = 622.37, orthorhombic, space group *Pbcm*, a = 11.069(2), b = 12.170(2), c = 25.275(5) Å, V = 3404.8(12) Å^3, Z = 4, D_{c} = 1.214 Mg m^{-3}, μ = 0.987 mm^{-1}, scan type ω scans, θ range 1.6–22.50°. GoF on F^2 0.999 for 2287 unique observed data and 190 parameters, R_1 = 0.0676, wR_2 = 0.1096. **2**: C$_{72}$H$_{98}$Cl$_4$In$_2$, M = 1334.94, orthorhombic, space group *Cmca*, a = 25.6540(13), b = 21.2270(11), c = 12.7609(6) Å, V = 6949.0(6) Å^3, Z = 4, D_{c} = 1.276 Mg m^{-3}, μ = 0.855 mm^{-1}, scan type ϕ and ω scans, θ range 1.92–29.00°. GOF on F^2 1.058 for 4706 unique observed data and 203 parameters, R_1 = 0.0418, wR_2 = 0.0691. **3**·2C$_6$H$_6$: C$_{84}$H$_{112}$Cl$_2$Ga$_2$O$_2$, M = 1364.08, monoclinic, space group $P2_1/c$, a = 14.2605(10), b = 15.9037(10), c = 18.0905(12) Å, β = 108.613(2)°, V = 3888.2(4) Å^3, Z = 2, D_{c} = 1.165 Mg m^{-3}, μ = 0.805 mm^{-1}, scan type ϕ and ω scans, θ range 1.75–22.50°. GOF on F^2 1.017 for 5074 unique observed data and 419 parameters, R_1 = 0.0620, wR_2 = 0.1037.

CCDC 182/1347. See http://www.rsc.org/suppdata/cc/1999/1805/ for crystallographic files in .cif format.

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