# Crystal structure of *catena*-hexakis($\mu$-4-methylbenzoato)didysprosium(III), $Dy_2(C_8H_7O_2)_6$

X. Li*[I], Z.-Y. Zhang[I], D.-Y. Wang[II] and Y.-Q. Zou[III]

[I] Capital Normal University, Department of Chemistry, Beijing, 100037 P. R. China
[II] Northwest University, Department of Chemistry, Xian, 710069 P. R. China
[III] Beijing Normal University, Department of Chemistry, Beijing, 100875 P. R. China

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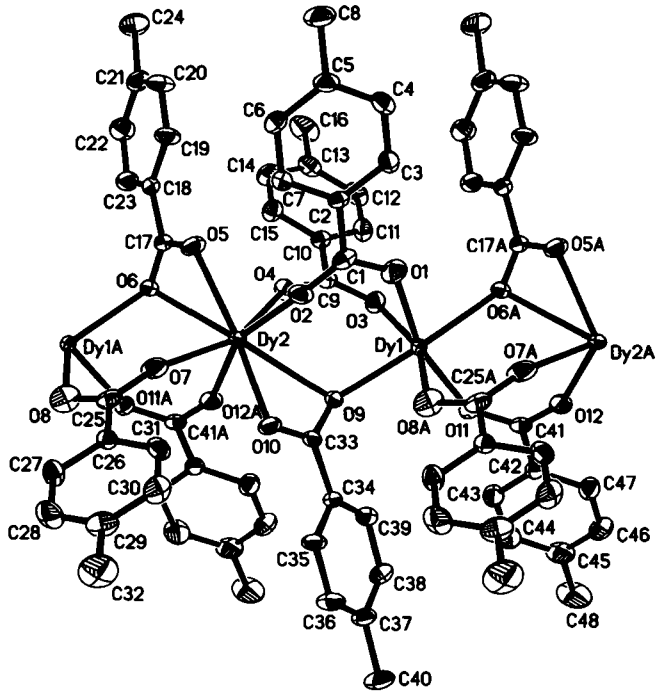


**Abstract**

$C_{48}H_{42}Dy_2O_{12}$, triclinic, $P\bar{1}$ (no. 2), $a = 8.155(2)$ Å, $b = 12.375(4)$ Å, $c = 22.168(6)$ Å, $\alpha = 92.626(4)°$, $\beta = 95.868(4)°$, $\gamma = 90.261(4)°$, $V = 2223.0$ Å$^3$, $Z = 2$, $R_{gt}(F) = 0.033$, $wR_{ref}(F^2) = 0.098$, $T = 293$ K.

**Source of material**

4-Methylbenzoic acid (1.5 mmol, 204 mg) was dissolved in ethanol (20 mL) and the pH of the solution was adjusted in the range 5-6 by using 2 M NaOH solution. The ethanol solution of $DyCl_3 \cdot 6H_2O$ (0.5 mmol, 189 mg) was successively dropped in. The mixture was heated under reflux with stirring for 2 h and then kept in the flask for six days. Crystals of the title compound obtained were used for the X-ray diffraction analysis.

Elemental analysis: found – C, 51.00 %; H, 4.05 %; calc. for $C_{48}H_{42}Dy_2O_{12}$ – C, 50.76 %; H, 3.73.

**Discussion**

Lanthanide carboxylate complexes display a variety of structural types, in which carboxylate groups link lanthanide ions in many coordination modes to form polynuclear compounds and one-, two- or three-dimensional coordination polymers.

In the title complex, there exist two different coordination environments of Dy(III) ions. Dy1 ion is coordinated by six O atoms (O1, O3, O9, O11, O6A and O8A) and Dy2 ion is surrounded by eight O atoms (O2, O4, O5, O6, O7, O9, O10 and O12A), both from six 4-methylbenzoato groups. Coordination number of lanthanide ions in most lanthanide carboxylate complexes is eight or nine, whereas the complexes with six ligands are rare. Dy1 and Dy2 ions are connected to each other through two 4-methylbenzoato groups in bidentate bridging and one in tridentate bridging mode to form a dimeric subunit, $[Dy(CH_3C_6H_4COO)_3]_2$. These subunits are linked to form an infinite one-dimensional chain by bridging of 4-methylbenzoato groups. O1-C1-O2 and O3-C9-O4 groups are in bidentate bridging, in which two O atoms coordinate two different Dy ions to form a bidentate bridge. O9-C33-O10 groups are in bridging-chelating mode, in which two O atoms chelate one Dy ion and one of them also simultaneously links another Dy ion to form a tridentate bridge. The distances between two neighboring Dy ions are 4.121 Å and 4.181 Å, respectively, and the angle Dy2–Dy1–Dy2A is 158.4°.

The crystal structure and coordination modes of carboxylate groups are different from those of Eu(*p*-$CH_3C_6H_4COO)_3$ [1] where carboxylate groups only adopt one bridging-chelating mode. The coordination environment of each Eu(III) ion is chemically equivalent, with eight coordinated O atoms, but unlike Nd(III) and Tb(III) [2] coordination polymers with 3-methylbenzoato. The structure of Nd(*m*-$CH_3C_6H_4COO)_3$ complex is similar to Eu(*p*-$CH_3C_6H_4COO)_3$. In Tb(*p*-$CH_3C_6H_4COO)_3$ complex, Tb ions are linked by two 3-methylbenzoato groups in chelating-bridging and one 3-methylbenzoato in bridging. It is obvious that the structural difference arises from different positions of the methyl group in the benzene ring. When a chelating ligand, 1,10-phenanthroline (phen) or 2,2′-bipyridine (bpy), was introduced to generate new mixed-ligand complexes, dinuclear molecules unlike the title complex were formed, such as Eu(*p*-$CH_3C_6H_4COO)_3$(phen) [3] and $Sm_2$(*p*-$CH_3C_6H_4COO)_6$(bpy)$_2$ [4].

Dy1—O bond lengths range from 2.247(4) Å to 2.307(4) Å, with an average distance 2.278(4) Å and Dy2—O bond lengths are in the range from 2.2593(5) Å to 2.702(4) Å, with an average distance of 2.394(4) Å. The distances of Dy1—O are shorter than those of Dy2—O, which are attributable to different coordination environments of the two Dy(III) ions. The O5–C17–O6 and O9–C33–O10 groups chelate Dy2 ions with two O atoms forming an unstable four-membered ring, resulting in longer Dy—O distances, $d$(Dy2—O6) = 2.702(4) Å and $d$(Dy2—O9) = 2.652(4) Å.

* Correspondence author (e-mail: zouyq@263.net)

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | yellow prism, size 0.12 × 0.20 × 0.26 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 33.98 $cm^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 50.02° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 11681, 7817 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 6473 |
| $N(param)_{refined}$: | 564 |
| Programs: | SHELXS-97 [5], SHELXL-97 [6] |

**Table 2.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(3) | 2*i* | 0.4315 | 0.6716 | 0.0592 | 0.051 |
| H(4) | 2*i* | 0.4214 | 0.6502 | –0.0447 | 0.061 |
| H(6) | 2*i* | 0.8920 | 0.5583 | –0.0293 | 0.053 |
| H(7) | 2*i* | 0.9040 | 0.5820 | 0.0749 | 0.046 |
| H(8A) | 2*i* | 0.6818 | 0.6461 | –0.1342 | 0.100 |
| H(8B) | 2*i* | 0.5348 | 0.5645 | –0.1328 | 0.100 |
| H(8C) | 2*i* | 0.7166 | 0.5228 | –0.1266 | 0.100 |
| H(11) | 2*i* | 0.6040 | 1.0980 | 0.2307 | 0.053 |
| H(12) | 2*i* | 0.6403 | 1.2649 | 0.1944 | 0.064 |
| H(14) | 2*i* | 1.0637 | 1.1684 | 0.1360 | 0.065 |
| H(15) | 2*i* | 1.0297 | 1.0024 | 0.1736 | 0.053 |
| H(16A) | 2*i* | 0.7865 | 1.3909 | 0.1441 | 0.121 |
| H(16B) | 2*i* | 0.8911 | 1.3405 | 0.0945 | 0.121 |
| H(16C) | 2*i* | 0.9779 | 1.3802 | 0.1581 | 0.121 |
| H(19) | 2*i* | 1.1049 | 0.7832 | 0.0426 | 0.057 |
| H(20) | 2*i* | 1.1359 | 0.9058 | –0.0307 | 0.067 |
| H(22) | 2*i* | 1.4175 | 1.0928 | 0.0901 | 0.069 |
| H(23) | 2*i* | 1.3952 | 0.9669 | 0.1626 | 0.056 |
| H(24A) | 2*i* | 1.1968 | 1.1454 | –0.0260 | 0.123 |
| H(24B) | 2*i* | 1.2866 | 1.0601 | –0.0652 | 0.123 |
| H(24C) | 2*i* | 1.3896 | 1.1381 | –0.0179 | 0.123 |
| H(27) | 2*i* | 1.5705 | 0.3819 | 0.3090 | 0.055 |
| H(28) | 2*i* | 1.5471 | 0.2039 | 0.3316 | 0.072 |
| H(30) | 2*i* | 1.0607 | 0.2145 | 0.2871 | 0.065 |
| H(31) | 2*i* | 1.0840 | 0.3915 | 0.2642 | 0.054 |
| H(32A) | 2*i* | 1.2941 | 0.0624 | 0.3676 | 0.134 |
| H(32B) | 2*i* | 1.3748 | 0.0309 | 0.3081 | 0.134 |
| H(32C) | 2*i* | 1.1829 | 0.0416 | 0.3062 | 0.134 |
| H(35) | 2*i* | 0.8819 | 0.4282 | 0.3864 | 0.052 |
| H(36) | 2*i* | 0.8320 | 0.3751 | 0.4816 | 0.064 |
| H(38) | 2*i* | 0.6400 | 0.6648 | 0.5169 | 0.063 |
| H(39) | 2*i* | 0.6800 | 0.7158 | 0.4207 | 0.052 |
| H(40A) | 2*i* | 0.6416 | 0.4173 | 0.5701 | 0.106 |
| H(40B) | 2*i* | 0.6517 | 0.5393 | 0.5927 | 0.106 |
| H(40C) | 2*i* | 0.8122 | 0.4697 | 0.5948 | 0.106 |
| H(43) | 2*i* | 0.4774 | 0.8652 | 0.4573 | 0.059 |
| H(44) | 2*i* | 0.4358 | 0.9229 | 0.5549 | 0.080 |
| H(46) | 2*i* | –0.0431 | 0.8631 | 0.5194 | 0.078 |
| H(47) | 2*i* | –0.0059 | 0.8072 | 0.4232 | 0.059 |
| H(48A) | 2*i* | 0.0587 | 0.9665 | 0.6128 | 0.159 |
| H(48B) | 2*i* | 0.2480 | 0.9905 | 0.6267 | 0.159 |
| H(48C) | 2*i* | 0.1773 | 0.8758 | 0.6377 | 0.159 |

**Table 3.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Dy(1) | 2*i* | 0.51832(3) | 0.74372(2) | 0.26301(1) | 0.0217(1) | 0.0282(2) | 0.0252(2) | –0.0014(1) | 0.0034(1) | 0.0035(1) |
| Dy(2) | 2*i* | 1.01599(3) | 0.69076(2) | 0.24253(1) | 0.0272(2) | 0.0268(2) | 0.0223(2) | 0.0007(1) | 0.0011(1) | 0.0055(1) |
| O(1) | 2*i* | 0.5544(6) | 0.6908(4) | 0.1670(2) | 0.058(3) | 0.072(3) | 0.037(3) | 0.001(3) | 0.015(2) | –0.011(2) |
| O(2) | 2*i* | 0.8056(5) | 0.6230(4) | 0.1770(2) | 0.047(3) | 0.051(3) | 0.031(2) | –0.011(2) | –0.007(2) | 0.004(2) |
| O(3) | 2*i* | 0.6527(5) | 0.9000(3) | 0.2491(2) | 0.036(2) | 0.033(2) | 0.054(3) | –0.007(2) | 0.009(2) | 0.009(2) |
| O(4) | 2*i* | 0.9016(5) | 0.8553(3) | 0.2248(2) | 0.041(2) | 0.029(2) | 0.064(3) | 0.019(2) | 0.013(2) | 0.022(2) |
| O(5) | 2*i* | 1.1052(5) | 0.7144(4) | 0.1457(2) | 0.050(3) | 0.049(3) | 0.034(2) | –0.013(2) | 0.009(2) | 0.010(2) |
| O(6) | 2*i* | 1.2771(5) | 0.8044(3) | 0.2126(2) | 0.033(2) | 0.043(2) | 0.028(2) | 0.008(2) | 0.003(2) | 0.010(2) |
| O(7) | 2*i* | 1.2235(6) | 0.5702(4) | 0.2462(2) | 0.083(4) | 0.056(3) | 0.063(3) | 0.037(3) | 0.037(3) | 0.035(3) |
| O(8) | 2*i* | 1.4859(6) | 0.5654(4) | 0.2807(2) | 0.075(4) | 0.037(3) | 0.076(4) | –0.021(2) | 0.011(3) | 0.004(3) |
| O(9) | 2*i* | 0.7704(4) | 0.6999(3) | 0.3123(2) | 0.026(2) | 0.039(2) | 0.032(2) | –0.002(2) | –0.001(2) | 0.012(2) |
| O(10) | 2*i* | 0.9287(5) | 0.5581(3) | 0.3062(2) | 0.051(3) | 0.042(2) | 0.038(2) | 0.007(2) | 0.016(2) | 0.016(2) |
| O(11) | 2*i* | 0.4058(5) | 0.7983(4) | 0.3485(2) | 0.036(2) | 0.054(3) | 0.028(2) | 0.000(2) | 0.007(2) | 0.001(2) |
| O(12) | 2*i* | 0.1383(5) | 0.7629(4) | 0.3319(2) | 0.043(3) | 0.072(3) | 0.024(2) | –0.014(2) | –0.004(2) | 0.000(2) |
| C(1) | 2*i* | 0.6780(7) | 0.6488(5) | 0.1448(3) | 0.047(4) | 0.027(3) | 0.032(3) | –0.016(3) | 0.011(3) | –0.006(3) |
| C(2) | 2*i* | 0.6691(7) | 0.6302(4) | 0.0778(2) | 0.035(3) | 0.030(3) | 0.022(3) | –0.006(2) | 0.004(2) | 0.000(2) |
| C(3) | 2*i* | 0.5249(7) | 0.6492(5) | 0.0415(3) | 0.034(3) | 0.054(4) | 0.039(4) | 0.004(3) | 0.002(3) | –0.001(3) |
| C(4) | 2*i* | 0.5184(8) | 0.6353(6) | –0.0209(3) | 0.042(4) | 0.071(5) | 0.037(4) | 0.005(3) | –0.009(3) | 0.001(3) |
| C(5) | 2*i* | 0.6546(8) | 0.5993(6) | –0.0484(3) | 0.053(4) | 0.054(4) | 0.024(3) | –0.005(3) | 0.000(3) | –0.001(3) |
| C(6) | 2*i* | 0.7986(8) | 0.5809(5) | –0.0116(3) | 0.043(4) | 0.056(4) | 0.035(4) | 0.006(3) | 0.015(3) | –0.003(3) |
| C(7) | 2*i* | 0.8065(7) | 0.5955(5) | 0.0510(3) | 0.034(3) | 0.048(4) | 0.035(3) | 0.002(3) | 0.003(3) | 0.009(3) |
| C(8) | 2*i* | 0.646(1) | 0.5816(7) | –0.1168(3) | 0.077(5) | 0.096(7) | 0.027(4) | –0.011(5) | 0.006(4) | 0.001(4) |
| C(9) | 2*i* | 0.7883(7) | 0.9211(5) | 0.2281(3) | 0.035(3) | 0.030(3) | 0.030(3) | –0.012(3) | –0.002(2) | 0.002(2) |
| C(10) | 2*i* | 0.8109(7) | 1.0301(4) | 0.2052(3) | 0.036(3) | 0.027(3) | 0.031(3) | –0.007(2) | 0.003(2) | 0.000(2) |
| C(11) | 2*i* | 0.6974(8) | 1.1117(5) | 0.2114(3) | 0.037(3) | 0.030(3) | 0.068(5) | 0.003(3) | 0.006(3) | 0.012(3) |
| C(12) | 2*i* | 0.7193(8) | 1.2121(5) | 0.1897(3) | 0.051(4) | 0.034(4) | 0.072(5) | 0.007(3) | –0.005(4) | 0.013(3) |
| C(13) | 2*i* | 0.8566(8) | 1.2361(5) | 0.1609(3) | 0.050(4) | 0.040(4) | 0.059(5) | –0.013(3) | –0.012(3) | 0.017(3) |
| C(14) | 2*i* | 0.9702(9) | 1.1547(6) | 0.1553(3) | 0.051(4) | 0.056(5) | 0.058(5) | –0.015(3) | 0.012(3) | 0.016(4) |
| C(15) | 2*i* | 0.9493(8) | 1.0547(5) | 0.1774(3) | 0.044(4) | 0.034(3) | 0.057(4) | –0.002(3) | 0.014(3) | 0.010(3) |
| C(16) | 2*i* | 0.880(1) | 1.3470(6) | 0.1373(4) | 0.084(6) | 0.048(5) | 0.107(7) | –0.017(4) | –0.018(5) | 0.041(5) |
| C(17) | 2*i* | 1.2103(7) | 0.7886(5) | 0.1579(3) | 0.033(3) | 0.042(3) | 0.026(3) | 0.004(3) | 0.006(2) | 0.007(3) |
| C(18) | 2*i* | 1.2440(7) | 0.8642(5) | 0.1110(3) | 0.030(3) | 0.039(3) | 0.030(3) | 0.005(2) | 0.006(2) | 0.008(3) |
| C(19) | 2*i* | 1.1680(8) | 0.8456(6) | 0.0524(3) | 0.058(4) | 0.049(4) | 0.037(4) | –0.005(3) | –0.003(3) | 0.021(3) |
| C(20) | 2*i* | 1.1858(9) | 0.9194(6) | 0.0086(3) | 0.073(5) | 0.063(5) | 0.032(4) | –0.001(4) | –0.004(3) | 0.018(3) |

**Table 3.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| C(21) | 2*i* | 1.2786(9) | 1.0149(6) | 0.0226(4) | 0.057(4) | 0.052(4) | 0.057(5) | 0.006(4) | 0.021(4) | 0.028(4) |
| C(22) | 2*i* | 1.3545(9) | 1.0305(6) | 0.0801(3) | 0.057(4) | 0.056(5) | 0.060(5) | −0.016(4) | 0.003(4) | 0.019(4) |
| C(23) | 2*i* | 1.3397(8) | 0.9553(6) | 0.1241(3) | 0.045(4) | 0.055(4) | 0.040(4) | −0.010(3) | 0.003(3) | 0.011(3) |
| C(24) | 2*i* | 1.289(1) | 1.0973(8) | −0.0262(4) | 0.090(7) | 0.088(7) | 0.076(6) | −0.001(5) | 0.020(5) | 0.055(6) |
| C(25) | 2*i* | 1.3469(8) | 0.5211(5) | 0.2690(3) | 0.062(4) | 0.028(3) | 0.031(3) | 0.006(3) | 0.020(3) | −0.001(3) |
| C(26) | 2*i* | 1.3304(7) | 0.4048(4) | 0.2823(3) | 0.035(3) | 0.027(3) | 0.031(3) | 0.003(2) | 0.003(2) | 0.002(2) |
| C(27) | 2*i* | 1.4677(8) | 0.3479(5) | 0.3038(3) | 0.034(3) | 0.035(3) | 0.069(5) | −0.002(3) | 0.001(3) | 0.009(3) |
| C(28) | 2*i* | 1.4531(9) | 0.2414(6) | 0.3177(4) | 0.061(5) | 0.039(4) | 0.079(6) | 0.014(3) | 0.000(4) | 0.014(4) |
| C(29) | 2*i* | 1.301(1) | 0.1882(5) | 0.3114(3) | 0.079(5) | 0.031(3) | 0.058(5) | −0.008(3) | 0.011(4) | 0.010(3) |
| C(30) | 2*i* | 1.1643(9) | 0.2474(6) | 0.2912(3) | 0.051(4) | 0.045(4) | 0.065(5) | −0.021(3) | 0.006(3) | 0.001(4) |
| C(31) | 2*i* | 1.1781(8) | 0.3535(5) | 0.2773(3) | 0.038(3) | 0.051(4) | 0.043(4) | −0.002(3) | −0.008(3) | 0.009(3) |
| C(32) | 2*i* | 1.287(1) | 0.0700(6) | 0.3245(5) | 0.115(8) | 0.043(5) | 0.114(8) | −0.006(5) | 0.022(6) | 0.025(5) |
| C(33) | 2*i* | 0.8294(7) | 0.6118(5) | 0.3344(3) | 0.028(3) | 0.037(3) | 0.032(3) | −0.004(2) | −0.001(2) | 0.014(3) |
| C(34) | 2*i* | 0.7879(7) | 0.5786(5) | 0.3938(3) | 0.030(3) | 0.042(3) | 0.028(3) | −0.002(2) | 0.002(2) | 0.013(3) |
| C(35) | 2*i* | 0.8324(8) | 0.4760(5) | 0.4125(3) | 0.054(4) | 0.045(4) | 0.032(4) | 0.003(3) | 0.005(3) | 0.017(3) |
| C(36) | 2*i* | 0.8034(9) | 0.4444(6) | 0.4699(3) | 0.072(5) | 0.048(4) | 0.042(4) | 0.004(4) | 0.007(4) | 0.022(3) |
| C(37) | 2*i* | 0.7333(8) | 0.5140(6) | 0.5095(3) | 0.054(4) | 0.066(5) | 0.027(4) | −0.009(4) | 0.004(3) | 0.013(3) |
| C(38) | 2*i* | 0.6879(8) | 0.6167(6) | 0.4905(3) | 0.054(4) | 0.071(5) | 0.035(4) | 0.004(4) | 0.018(3) | 0.007(4) |
| C(39) | 2*i* | 0.7133(8) | 0.6478(5) | 0.4330(3) | 0.045(4) | 0.046(4) | 0.040(4) | 0.007(3) | 0.007(3) | 0.015(3) |
| C(40) | 2*i* | 0.707(1) | 0.4822(8) | 0.5724(3) | 0.085(6) | 0.097(7) | 0.033(4) | −0.006(5) | 0.014(4) | 0.023(4) |
| C(41) | 2*i* | 0.2627(7) | 0.7950(4) | 0.3657(3) | 0.045(3) | 0.025(3) | 0.027(3) | 0.002(2) | 0.004(3) | 0.006(2) |
| C(42) | 2*i* | 0.2387(7) | 0.8316(5) | 0.4284(3) | 0.036(3) | 0.036(3) | 0.030(3) | −0.001(2) | 0.003(2) | 0.004(3) |
| C(43) | 2*i* | 0.3714(8) | 0.8654(6) | 0.4693(3) | 0.041(4) | 0.072(5) | 0.032(4) | −0.001(3) | 0.003(3) | −0.009(3) |
| C(44) | 2*i* | 0.346(1) | 0.8996(7) | 0.5282(3) | 0.068(5) | 0.089(6) | 0.040(4) | 0.002(4) | −0.007(4) | −0.015(4) |
| C(45) | 2*i* | 0.193(1) | 0.8999(7) | 0.5480(3) | 0.085(6) | 0.077(6) | 0.033(4) | 0.028(5) | 0.016(4) | −0.001(4) |
| C(46) | 2*i* | 0.063(1) | 0.8643(7) | 0.5071(3) | 0.061(5) | 0.094(6) | 0.046(5) | 0.015(4) | 0.029(4) | 0.006(4) |
| C(47) | 2*i* | 0.0848(8) | 0.8310(6) | 0.4494(3) | 0.043(4) | 0.059(4) | 0.045(4) | 0.000(3) | 0.009(3) | −0.001(3) |
| C(48) | 2*i* | 0.167(1) | 0.937(1) | 0.6121(4) | 0.134(9) | 0.15(1) | 0.032(5) | 0.066(8) | 0.019(5) | −0.012(5) |

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