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#### Key indicators

Single-crystal X-ray study  
 $T = 120$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.042  
 $wR$  factor = 0.124  
Data-to-parameter ratio = 26.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

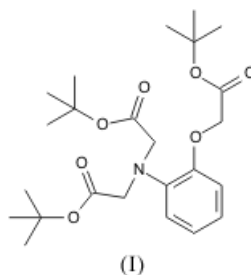
## Di-*tert*-butyl 2-(*tert*-butyloxycarbonyl-methoxy)phenyliminodiacetate

The single-crystal structure of the title compound,  $\text{C}_{24}\text{H}_{37}\text{NO}_7$ , which is a derivative of the commonly used chelating agent aminophenol triacetic acid, has been determined. The structure was solved in space group  $P\bar{1}$  with two molecules in the unit cell. As expected, the geometric conformation of this derivative of the free ligand differs extensively from that of the coordinated form.

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#### Comment

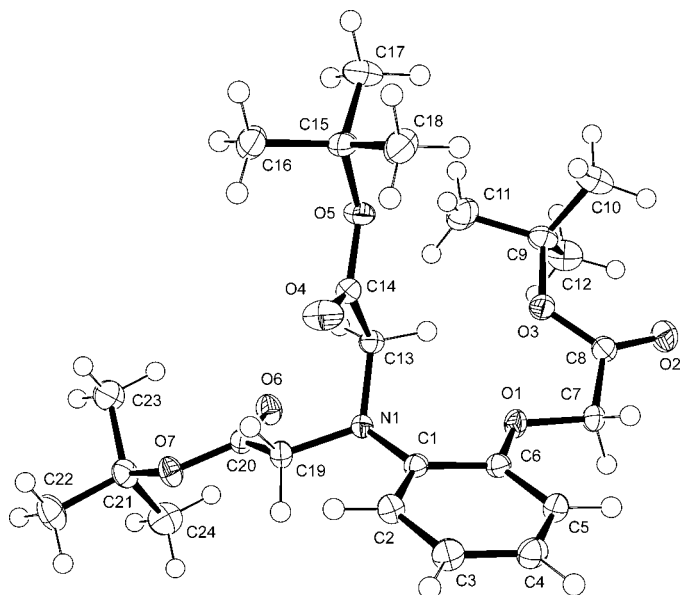
The title compound, (I), is a synthetic precursor to 2-aminophenol triacetic acid (APTA). APTA has been used as a chelating agent for earth metal species in aqueous solution. The ligand has demonstrated good selectivity for magnesium over calcium in intracellular domains. Derivatives of APTA have monitored magnesium cation levels within cells using techniques such as  $^{19}\text{F}$  NMR (Levy *et al.*, 1988) and fluorescence spectroscopy (Raju *et al.*, 1989; Otten *et al.*, 2001). We have reported magnesium sensors based on photo-induced electron transfer (PET) logic design which demonstrate enhanced fluorescence output in the presence of magnesium cations at intracellular concentrations (Desilva *et al.*, 1993).



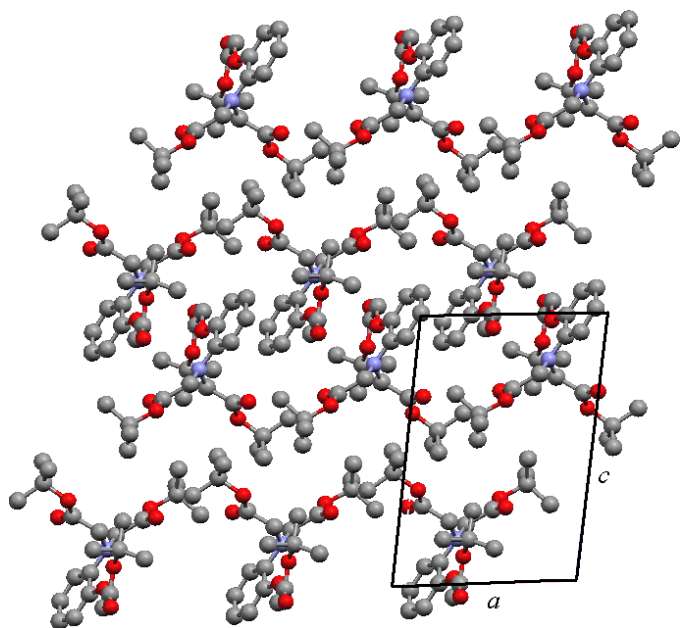
The single-crystal structure of the hexadentate APTA ligand coordinated with  $\text{Co}^{\text{II}}$  has been reported (Hualin *et al.*, 1990). In this structure, the central Co atom is bonded to the N atom and five O atoms, forming a distorted octahedron. However, the crystal structure of the uncoordinated tertiary butyl derivative, (I), differs significantly.

Fig. 1 shows the molecular structure of (I) and the atomic numbering used. All bond distances and angles are within the range of accepted values. The aromatic ring and atoms N1, O1 and C7 are coplanar, with an r.m.s. deviation of 0.026 Å. The maximum absolute deviation from the plane is 0.028 Å for C7. When coordinated to  $\text{Co}^{\text{II}}$ , the corresponding atoms in the APTA ligand are also coplanar.

The conformations of the two chains attached to the N atom are all-*trans*, with atoms N1, C13, O4, C14, O5, C15 and C17 coplanar in one chain, and atoms C22, C21, O7, O6 and C20 coplanar in the other. The r.m.s. deviations of the planes are



**Figure 1**  
The molecular structure of (I), showing the atomic numbering scheme and displacement ellipsoids drawn at the 50% probability level (ORTEP-3; Farrugia, 1997).



**Figure 2**  
Packing diagram for (I), viewed down the *b* axis, showing the layered packing (MERCURY; Bruno *et al.*, 2002).

0.011 and 0.031 Å, respectively. The angle between the two chain planes is 109.09 (5)°, with one chain extending below and the other above the plane of the aromatic ring.

There is a *gauche* bond between atoms C7 and C8 in the chain attached to the ether O atom. This deviation from the all-*trans* conformation is expressed in the deviation of the O1—C7—C8—O3 torsion angle [3.10 (11)°] from the all-*trans* values seen in both of the other chains.

The three-dimensional packing of molecules is illustrated in Fig. 2. A layered structure is formed, with the aromatic rings

and the chains attached to the ether O atom packing in a single layer, and the chains attached to the N atom forming the other. These layers are parallel to and extend infinitely in the *ab* plane.

In this crystal structure, no intermolecular  $\pi$ – $\pi$  interactions are observed, and the centroid-to-centroid distance between neighbouring aromatic rings is 4.360 (3) Å.

## Experimental

The title compound was prepared by the addition of anhydrous sodium carbonate (29 g, 106 mmol) and anhydrous sodium iodide (1.7 g, 11.5 mmol) to a solution of 2-aminophenol (5 g, 45.8 mmol) in dry acetonitrile (100 ml). Finally, *tert*-butyl bromomethylacetate (31.3 g, 160 mmol) was added. The mixture was refluxed overnight under an inert atmosphere of nitrogen. The resulting solution was filtered and the solvent evaporated. The residue was first chromatographed on silica using a mixture of ethyl acetate and hexane. The product was then recrystallized from methanol affording 3.2 g of the product. Needle-shaped crystals of (I) (m.p. 339–340 K) were grown from methanol at room temperature.

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  1.40 (s, 18H, *tert*-butyl), 1.44 (s, 9H, *tert*-butyl), 4.05 (s, 4H, NCH<sub>2</sub>), 4.52 (s, 4H, OCH<sub>2</sub>), 6.81–6.92 (*m*, 4H, ArH); <sup>13</sup>C NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  28.0 (s, C-16, C-17, C-18, C-22, C-23, C-24), 28.1 (s, C-10, C-11, C-12), 66.6 (s, C-9, C-15, C-21), 80.1 (s, C-13, C-19), 81.9 (s, C-7), 114.3 (s, C-3), 119.6 (s, C-2), 121.9 (s, C-4), 122.0 (s, C-5), 139.5 (s, C-1), 149.6 (s, C-6), 168.3 (s, C-14, C-20), 170.5 (s, C-8).

## Crystal data

C <sub>24</sub> H <sub>37</sub> NO <sub>7</sub>	<i>Z</i> = 2
<i>M<sub>r</sub></i> = 451.55	<i>D<sub>x</sub></i> = 1.212 Mg m <sup>−3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> $\alpha$ radiation
<i>a</i> = 8.559 (5) Å	Cell parameters from 559 reflections
<i>b</i> = 11.192 (4) Å	$\theta$ = 2–31°
<i>c</i> = 13.689 (5) Å	$\mu$ = 0.09 mm <sup>−1</sup>
$\alpha$ = 106.03 (3)°	<i>T</i> = 120 (2) K
$\beta$ = 96.27 (4)°	Block, colourless
$\gamma$ = 96.89 (4)°	0.55 × 0.40 × 0.30 mm
<i>V</i> = 1237.2 (10) Å <sup>3</sup>	

## Data collection

Oxford Excalibur2 diffractometer	<i>R</i> <sub>int</sub> = 0.021
$\omega$ – $2\theta$ scans	$\theta$ <sub>max</sub> = 31.9°
Absorption correction: none none	<i>h</i> = −12 → 10
12422 measured reflections	<i>k</i> = −16 → 16
7546 independent reflections	<i>l</i> = −18 → 20
5991 reflections with <i>I</i> > 2 $\sigma$ ( <i>I</i> )	

## Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.0751P]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	( $\Delta/\sigma$ ) <sub>max</sub> = 0.001
<i>S</i> = 1.13	$\Delta\rho$ <sub>max</sub> = 0.43 e Å <sup>−3</sup>
7546 reflections	$\Delta\rho$ <sub>min</sub> = −0.23 e Å <sup>−3</sup>
289 parameters	
H-atom parameters constrained	

All H atoms were placed in calculated positions (C–H = 0.95–0.99 Å), and refined using a riding model (*U*<sub>iso</sub> = 1.2 or 1.5 times *U*<sub>eq</sub>(parent atom), with rotation in the case of the terminal methyl groups).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows*

(Farrugia, 1997) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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