

1 and of the carbonyl ligands occurs in the reaction of 2 with dppe (dppe =  $\text{Ph}_2\text{PCH}_2\text{CH}_2\text{PPh}_2$ ), and the product is the known  $[\text{Rh}(\text{dppe})_2]\text{PF}_6$ . In the formation of 6 and 7 an excess of ligand must be avoided. Complexes 6 and 7 are air stable, yellow-brown solids, soluble in acetone and chlorinated solvents; their conductivities, in acetone solution, are in the range found for 1:2 electrolytes. Apart from the shift in the carbonyl stretching frequency, the IR spectra of 6 and 7 closely resemble the spectrum of 5. The proton NMR spectra of 6 and 7 show a chemical shift difference between  $\alpha$  and  $\beta$  protons of ligand 1 comparable with that of complex 5. By analogy to 3-5 we assume these complexes (1) are  $\eta^5$ -coordinated.

The solvent complex  $[\text{Rh}(\text{C}_5\text{Me}_5)(\text{acetone})_3][\text{PF}_6]_2$ <sup>23</sup> easily reacts with 1 to give the sandwich-type rhodium(III) complex  $[\text{Rh}(\text{C}_5\text{Me}_5)(\text{Ph}_3\text{PC}_5\text{H}_4)][\text{PF}_6]_2$  (8) as a yellow-brown solid. In the proton NMR spectrum of 8 the resonances of  $\alpha$  and  $\beta$  protons of coordinated  $\text{Ph}_3\text{PC}_5\text{H}_4$  are not distinguishable since they are split into a multiplet at  $\tau$  3.78. A zero chemical shift difference between  $\alpha$  and  $\beta$  protons was observed<sup>24</sup> for the cationic complex  $[\text{Pd}(\eta^3\text{-allyl})(\text{Ph}_3\text{PC}_5\text{H}_4)]\text{BF}_4$ , and the chemical shift difference of the  $\alpha$  and  $\beta$  protons in the analogous  $[\text{Pd}(\eta^3\text{-Ph}_3\text{PC}_5\text{H}_4)(\text{Ph}_3\text{PC}_5\text{H}_4)][\text{BF}_4]_2$  was attributed to an anisotropy of the phenyl rings on the triphenylphosphonium allylide ligand. The high formal oxidation state of the metal in complex 8 probably determines the lack of difference in the chemical shift. However, the <sup>13</sup>C NMR chemical shifts of 8 fall in the range of the described complexes; the major difference is observed, as expected, in the values of the coupling constants between <sup>103</sup>Rh and <sup>13</sup>C.

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**Registry No.** 1, charged form, 29473-30-1; 1, uncharged form, 2224-32-0; 2,  $\text{PF}_6$  salt, 77589-85-6; 2,  $\text{BPh}_4$  salt, 77589-86-7; 3,  $\text{PF}_6$  salt, 77589-88-9; 3,  $\text{BPh}_4$  salt, 77589-89-0; 4,  $\text{PF}_6$  salt, 77589-91-4; 4,  $\text{BPh}_4$  salt, 77589-92-5; 5,  $\text{PF}_6$  salt, 77589-94-7; 5,  $\text{BPh}_4$  salt, 77589-95-8; 6, 77589-97-0; 7, 77611-44-0; 8, 77589-99-2;  $[\text{Rh}(\text{C}-\text{O})_2\text{Cl}]_2$ , 14404-25-2;  $[\text{Rh}(\text{1,5-}\text{C}_6\text{H}_4)_2\text{Cl}]_2$ , 12092-47-6;  $[\text{Rh}(\text{C}_7\text{H}_8)\text{Cl}]_2$ , 12257-42-0;  $[\text{Rh}(\text{C}_5\text{Me}_5)\text{Cl}]_2$ , 12354-85-7;  $[\text{Rh}(\text{C}_5\text{Me}_5)(\text{acetone})_3][\text{PF}_6]_2$ , 60936-90-5.

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### Synthesis and Structure of Hexakis(*tert*-butyl isocyanide)vanadium(II) Hexacarbonylvanadate(-I)

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We recently reported the synthesis of  $[\text{V}(\text{CN-}t\text{-Bu})_6]^{2+}$  by reduction of vanadium(III) chloride in ethanol.<sup>1</sup> The hexafluorophosphate salt of this homoleptic vanadium isocyanide complex was characterized by elemental analysis and infrared

spectroscopy. In the present study, the  $[\text{V}(\text{CN-}t\text{-Bu})_6]^{2+}$  cation was obtained from  $[\text{V}(\text{CO})_6]^-$  by oxidation and addition of *tert*-butyl isocyanide. This convenient new route parallels that used previously<sup>2</sup> to obtain the homoleptic seven-coordinate isocyanide complexes of molybdenum(II) and tungsten(II). We also describe the crystal and molecular structure of  $[\text{V}(\text{CN-}t\text{-Bu})_6][\text{V}(\text{CO})_6]_2$ .

### Experimental Section and Results

**Synthetic Work.** All compounds were handled under an atmosphere of dry nitrogen with use of Schlenk techniques or in a Vacuum Atmosphere drybox. *tert*-Butyl isocyanide,<sup>3</sup> tetraethylammonium hexacarbonylvanadate(-I),<sup>4</sup> trichlorotris(*tert*-butyl isocyanide)vanadium(III),<sup>1</sup> and iodobenzene dichloride ( $\text{PhICl}_2$ )<sup>5</sup> were prepared by literature methods. All other starting materials were commercially available. Solvents were distilled from appropriate drying agents under nitrogen and degassed immediately before use. Chemical analyses were performed by Galbraith Laboratories, Knoxville, Tn.

### Preparation of Hexakis(*tert*-butyl isocyanide)vanadium(II) Hexacarbonylvanadate(-I). Method 1.

A slurry of 1.1 g (3.1 mmol) of tetraethylammonium hexacarbonylvanadate(-I) in 15 mL of ethanol and 5 mL (48 mmol) of *tert*-butyl isocyanide was combined with a slurry of 0.473 g (1.72 mmol) of iodobenzene dichloride in 15 mL of ethanol. A vigorous reaction occurred with the evolution of gas, and the product precipitated as an orange solid. The reaction mixture was stirred until all of the yellow, solid tetraethylammonium hexacarbonylvanadate(-I) was consumed (0.5 h). The crude product was collected by filtration, washed with a small volume of ethanol, and vacuum-dried. The product was recrystallized from 10 mL of methylene chloride by layering on 20 mL of diethyl ether. The yield of red crystalline product was 0.73 g (73%). Anal. Calcd for  $\text{C}_{42}\text{H}_{54}\text{N}_6\text{O}_{12}\text{V}_2$ : C, 51.07; H, 5.51; N, 8.51; O, 19.44; V, 15.47. Found: C, 51.16; H, 5.69; N, 8.50; O, 19.33 (by difference); V, 15.32. IR (Nujol mull): 2197 (s), 1850 (s, br), 1235 (w), 1200 (m), 1020 (w), 850 (w), 655 (s), 520 (w), 455 (w)  $\text{cm}^{-1}$ . <sup>1</sup>H NMR in  $\text{CD}_2\text{Cl}_2$  vs.  $\text{Me}_4\text{Si}$ :  $\delta$  5 ( $\Delta\nu_{1/2}$  = 250 Hz).

**Method 2.** A solution containing 0.20 g (0.49 mmol) of trichlorotris(*tert*-butyl isocyanide)vanadium(III) in 6 mL of ethanol and 4 mL (38 mmol) of *tert*-butyl isocyanide was syringed onto 0.37 g (1.1 mmol) of solid tetraethylammonium hexacarbonylvanadate(-I). The product precipitates as the tetraethylammonium hexacarbonylvanadate(-I) dissolves. Hexakis(*tert*-butyl isocyanide)vanadium(II) hexacarbonylvanadate(-I) (0.18 g, 38%) was collected by filtration and identified by its color, solubility properties, and infrared spectrum.

**Crystallographic Work.** While crystals of  $[\text{V}(\text{CN-}t\text{-Bu})_6][\text{V}(\text{CO})_6]_2$  could be examined briefly in air, diffracted intensity was lost over periods of hours or days unless the most scrupulous care was taken to exclude air at all stages. Crystals used in the structure determination were selected and mounted under an atmosphere of nitrogen in a glovebag containing  $\text{P}_2\text{O}_5$  to scavenge water and freshly reduced BASF catalyst to scavenge oxygen. Crystals were pushed into 0.5-mm thin-walled glass capillary tubes which had been prepurged in a drybox and were held in place with silicone grease. A first set of data was collected with use of graphite-monochromated Mo  $K\alpha$  radiation. Intensities from this crystal fell off rapidly at higher scattering angles, and data could be gathered only

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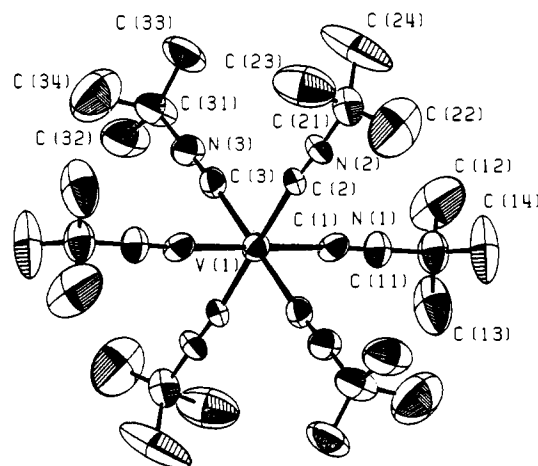
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**Table I.** Experimental Details of the X-ray Diffraction Study of Hexakis(*tert*-butyl isocyanide)vanadium(II) Hexacarbonylvanadate(−I)

(A) Crystal Parameters at 23 °C <sup>a</sup>		
$a = 10.741$ (1) Å	space group = $P2_1/n$	
$b = 23.122$ (5) Å	fw = 987.8	
$c = 12.142$ (5) Å	$Z = 2$	
$\beta = 100.50$ (3)°	calcd density = 1.11 g/cm <sup>3</sup>	
$V = 2965$ (2) Å <sup>3</sup>	obsd density = 1.12 (1) g/cm <sup>3</sup> <sup>b</sup>	
(B) Measurement and Treatment of Intensity Data		
	Mo data set	Cu data set
instrument	Enraf-Nonius CAD 4F $\kappa$ -geometry diffractometer	
$\lambda(K\alpha_1)$	0.709 30 Å	1.540 51 Å
$\lambda(K\alpha)$	0.710 73 Å	1.541 78 Å
wavelength selection	graphite monochromator	Ni filter
takeoff angle	2.0°	5.0°
detector aperture		
vertical	4.0 mm	5.0 mm
horizontal	(3.0 + tan $\theta$ )°	(3.0 + tan $\theta$ )°
cryst-detector dist	173 mm	173 mm
scan method	$\omega$ (cryst)– 2 $\theta$ (counter)	$\omega$ (cryst)– 2 $\theta$ (counter)
scan width	$\omega = (0.7 + 0.35 \tan \theta)$ °	$\omega = (1.0 + 0.15 \tan \theta)$ °
scan rate		
prescans	6.7°/min in $\theta$	4.0°/min in $\theta$
final scans	0.8–6.7°/min	1.0–4.0°/min, 0.7°/min fixed
max scan time	60 s	90 s, 120 s for fixed scans
scan range	2° < 2 $\theta$ < 40°	2° < 2 $\theta$ < 110°
bkg	moving crystal–moving detector, 25% added to scan width at both ends of each scan in both data sets	
check reflctns	(1,2,2), (3,4,–1), (0,9,3)	(1,2,2), (3,4,–1), (0,9,3)
time between checks	2500 s	2500 s, 3600 s for fixed scans
fall-off correction	4%, random	21%, smoothed decrease
total no. of data	2797	3770 plus 1100 higher angle duplicates at slow scans
data for $I > 2\sigma(I)$	1171	1167
(C) Data Reduction		
	Mo data set	Cu data set
redn to $F$ and to $\sigma(F)$	correction for bkgd, attenuator, Lorentz–polarization of monochromatized (for Mo) X-radiation, and absorption as described previously <sup>c</sup>	
abs correction		
cryst size	0.5 × 0.5 × 0.4 mm	0.15 × 0.45 × 0.33 mm
transmission	0.78–0.82 estimated,	0.27–0.53
coefficients	correction not made	
(D) Final Model in the Least Squares Refinement <sup>d</sup>		
	Mo data set	Cu data set
final $R$ value <sup>e</sup>	0.075	0.092
$R_w$	0.102	0.096
no. of observns	1171	1167
no. of parameters	287	287
weights	$w = 1/\sigma^2$ with $\sigma^2 = \sigma^2(\text{counts}) + 0.005F^2$	
highest peak in final difference map	0.35 e/Å <sup>3</sup>	0.53 e/Å <sup>3</sup>
amt C atom peak	35%	53%

<sup>a</sup> From a least-squares fit to the setting angles of 25 reflections with  $\theta$  in the range 10–28° with use of Cu  $K\alpha$  radiation. <sup>b</sup> By suspension in cyclohexane/carbon tetrachloride mixtures in the absence of air. <sup>c</sup> Reference 1. <sup>d</sup> Refinements were carried out with the use of G. Sheldrick's SHELX program. Anisotropic thermal parameters for V, C, and O atoms were used. Fixed H atom positions were recalculated after each cycle, with C–H = 0.95 Å and H atoms in staggered conformations. <sup>e</sup>  $R = \Sigma|\Delta|/\Sigma|F_O|$ ,  $R_w = (\Sigma w\Delta^2/\Sigma wF_O^2)$ , and  $\Delta = |F_O| - |F_C|$ .

<sup>a</sup> From a least-squares fit to the setting angles of 25 reflections with  $\theta$  in the range 10–28° with use of Cu  $K\alpha$  radiation. <sup>b</sup> By suspension in cyclohexane/carbon tetrachloride mixtures in the absence of air. <sup>c</sup> Reference 1. <sup>d</sup> Refinements were carried out with the use of G. Sheldrick's SHELX program. Anisotropic thermal parameters for V, C, and O atoms were used. Fixed H atom positions were recalculated after each cycle, with C–H = 0.95 Å and H atoms in staggered conformations. <sup>e</sup>  $R = \sum | \Delta | / \sum | F_o |$ ,  $R_w = (\sum w \Delta^2 / \sum w F_o^2)^{1/2}$ , and  $\Delta = | F_o | - | F_c |$ .



**Figure 1.** Structure of the  $[V(CN-t-Bu)_6]^{2+}$  cation showing the atom labeling scheme and 40% probability thermal ellipsoids. The vanadium atom is situated on an inversion center.

out to  $2\theta = 40^\circ$ . Even within this angular range, the majority of the intensities were measured to be less than background. An attempt was made to improve the crystallographic results by using Cu  $K\alpha$  radiation with freshly prepared and recrystallized samples. There seemed to be an even more rapid loss of diffraction intensity on exposure to Cu  $K\alpha$  radiation, however. In several batches examined, only one crystal was found to be of X-ray quality with relatively stable diffracted intensities. A complete set of data was obtained at room temperature with this crystal. Preliminary work at low temperatures indicated an encouraging increase in diffracted intensities at higher angles, but the intensities from one good crystal decreased dramatically over a period of a few days. The crystal system, data collected on two different crystals at room temperature, and data reduction are summarized in Table I.

The structure was solved by heavy-atom methods without difficulty, with use of the Mo  $K\alpha$  data. Least-squares refinement presented some problems. The thermal motions found for the light atoms were rather high, suggesting the need for an anisotropic model. The number of reflections above background was less than 1200, however, resulting in a 4:1 ratio of data to refined parameters. Convergence was finally achieved with use of both sets of data. Details of the final refinement model are given in Table I.

In order to test for systematic errors in the refinements with such a small set of observed data, we also carried out a refinement with use of all of the Mo  $K\alpha$  data, including zeros. The observed planes were weighted so that the average value of  $w\Delta^2$  varied by no more than a factor of 1.7:1 for various subsets of the data with different theta values and structure factor magnitudes. The  $R$  factor for this refinement was 0.21, and the parameters obtained did not differ significantly from those reported in this paper.

Averaged positional parameters for all atoms except hydrogen are given in Table II. Other information is available as supplementary material.<sup>6</sup>

## Discussion

A number of six-coordinate, homoleptic vanadium(II) cations are known with oxygen and nitrogen donor atom ligands. These complexes, of general formula  $[VL_6]X_2$ , have been prepared by reduction of V(III) or V(V) in the presence of the ligand (e.g.:  $L = H_2O$ ;  $X = \text{halide}, ClO_4^-$ )<sup>7</sup> or by the spontaneous disproportionation of  $V(CO)_6$  in a donor solvent

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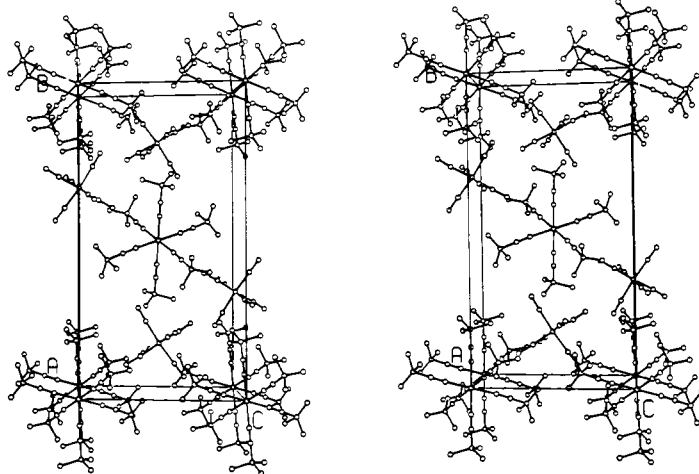


Figure 2. Crystal packing diagram of  $[\text{V}(\text{CN-}t\text{-Bu})_6][\text{V}(\text{CO})_6]_2$ .

(e.g.: L = acetone, acetaldehyde, DMF, methanol, others; X =  $\text{V}(\text{CO})_6^-$ ).<sup>8</sup> The disproportionation reactions of  $\text{V}(\text{CO})_6$  are especially interesting because of the variety of products formed and their dependence on reaction conditions. In nonpolar solvents vanadium(0) is stable; thus,  $\text{V}(\text{CO})_5\text{PPh}_3$  can be prepared from  $\text{V}(\text{CO})_6$  in cold  $\text{CH}_2\text{Cl}_2$ .<sup>4</sup> Donor solvents produce disproportionation to the  $[\text{VL}_6][\text{V}(\text{CO})_6]_2$  compounds mentioned above or to compounds of the formula  $[\text{VL}_4][\text{V}(\text{CO})_6]_2$  (e.g., L =  $\alpha$ -picoline,  $\text{OPPh}_3$ ,  $\text{Me}_2\text{SO}$ , others).<sup>8</sup> In one example of a complex of the latter type,  $[\text{V}(\text{THF})_4][\text{V}(\text{CO})_6]_2$ , the vanadium(II) atom is coordinated to two trans carbonyl groups through oxygen atoms.<sup>9</sup> This structure contains a linear V—C—O—V bridge.  $\text{V}(\text{CO})_6$  decomposes in aromatic solvents from which vanadium(I) compounds  $[\text{V}(\text{CO})_4(\text{arene})][\text{V}(\text{CO})_6]$  (arene = toluene, *p*-xylene, mesitylene, others)<sup>10</sup> and vanadium—vanadium bonded species,  $(\text{C}_6\text{H}_5)_2\text{V}_2(\text{CO})_4$ ,<sup>11</sup> have been isolated.

The synthesis of  $[\text{V}(\text{CN-}t\text{-Bu})_6][\text{V}(\text{CO})_6]_2$  by oxidation of  $\text{V}(\text{CO})_6^-$  in the presence of *tert*-butyl isocyanide is formally analogous to the  $\text{V}(0) \rightarrow \text{V}(\text{II}) + \text{V}(\text{I})$  disproportionation reactions discussed above. While all of the cited studies used freshly prepared  $\text{V}(\text{CO})_6$  as starting material, the present synthesis employs hexacarbonylvanadate(I) with 1 equiv of oxidant  $[\text{V}(\text{CO})_6^- + \frac{1}{2}\text{PhICl}_2]$ . The mechanism of the reaction does not necessarily involve generation of  $\text{V}(\text{CO})_6$  in situ followed by disproportionation; however, the product obtained would be that expected from such a disproportionation. This new route to the  $[\text{V}(\text{CN-}t\text{-Bu})_6]^{2+}$  cation has the advantage of using less expensive, more stable starting materials. The compound  $[\text{V}(\text{CN-}t\text{-Bu})_6][\text{V}(\text{CO})_6]_2$  has also been prepared by reduction of  $\text{V}(\text{CN-}t\text{-Bu})_3\text{Cl}_3$  with excess isocyanide followed by precipitation with the  $[\text{V}(\text{CO})_6]^-$  anion in a manner analogous to that previously reported for the preparation of  $[\text{V}(\text{CN-}t\text{-Bu})_6](\text{PF}_6)_2$ .

The crystal structure is composed of  $[\text{V}(\text{CN-}t\text{-Bu})_6]^{2+}$  cations lying on an inversion center at the origin of the unit cell and of  $[\text{V}(\text{CO})_6]^-$  anions situated in general positions. Figure 1 shows the octahedral cation and Figure 2 the unit cell packing diagram. Bond lengths and angles are given in Table III. The average V—C bond length in the cation is 2.10 (1) Å, which may be compared with the mean V—C distance of 2.161 (4) Å in  $\text{K}_4[\text{V}(\text{CN})_6]$ .<sup>12</sup> In the octahedral anion,

Table II. Final Positional Parameters for  $[\text{V}(\text{CN-}t\text{-Bu})_6][\text{V}(\text{CO})_6]_2^a$

Atom	x	y	z
V(1)	0.000	0.000	0.000
V(2)	0.2463(3)	0.1713(1)	0.4951(3)
C(4)	0.245(2)	0.242(1)	0.413(2)
C(5)	0.097(3)	0.195(1)	0.541(2)
C(6)	0.145(2)	0.131(1)	0.373(3)
C(7)	0.392(2)	0.149(1)	0.441(2)
C(8)	0.353(2)	0.207(1)	0.619(2)
C(9)	0.245(2)	0.106(1)	0.587(2)
O(4)	0.245(2)	0.285(1)	0.364(2)
O(5)	0.005(2)	0.212(1)	0.580(3)
O(6)	0.095(2)	0.107(1)	0.288(2)
O(7)	0.477(2)	0.132(1)	0.404(1)
O(8)	0.417(2)	0.231(1)	0.694(2)
O(9)	0.244(3)	0.066(1)	0.643(2)
C(1)	-0.124(2)	0.040(1)	0.091(1)
N(1)	-0.196(2)	0.063(1)	0.136(1)
C(11)	-0.207(3)	0.091(1)	0.189(2)
C(12)	-0.236(3)	0.102(2)	0.301(3)
C(13)	-0.395(3)	0.055(1)	0.186(4)
C(14)	-0.323(3)	0.148(1)	0.132(3)
C(2)	0.096(2)	0.080(1)	-0.004(2)
N(2)	0.139(2)	0.124(1)	-0.011(1)
C(21)	0.197(2)	0.179(1)	-0.017(2)
C(22)	0.121(4)	0.217(1)	-0.077(6)
C(23)	0.307(4)	0.172(2)	-0.072(3)
C(24)	0.250(5)	0.197(2)	0.105(3)
C(3)	0.126(2)	-0.025(1)	0.145(1)
N(3)	0.189(1)	-0.041(1)	0.225(1)
C(31)	0.274(3)	-0.062(1)	0.323(2)
C(32)	0.239(3)	-0.115(1)	0.357(2)
C(33)	0.331(3)	-0.018(1)	0.393(2)
C(34)	0.397(4)	-0.087(2)	0.269(3)

<sup>a</sup> Coordinates are the unweighted average of values from the two data sets. Numbers in parentheses are the average errors in the last significant digit(s).

the average V—C distance is 1.89 (2) Å, or 1.92 (2) Å after a riding correction for thermal motion is applied. These results are comparable within error limits to the value of 1.931 (9) Å found in  $[(\text{Ph}_3\text{P})_2\text{N}][\text{V}(\text{CO})_6]$ .<sup>13</sup> It is noteworthy that the rather wide variation in C—C distances in the *tert*-butyl groups was reproduced faithfully in each of the two completely independent structure determinations. This result indicates similar systematic errors in the data or the model for the two crystals. There were insufficient data to explore disordered models for the *tert*-butyl groups.

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