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N,*N*-Diethylpyrrolidinium pentachloro(tetrahydrofuran)zirconate(IV)

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Key indicators

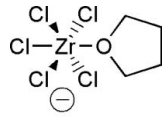
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.108
Data-to-parameter ratio = 21.4

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

Both ions of the title compound, $(C_8H_{18}N)[ZrCl_5(C_4H_8O)]$, exhibit approximate C_s symmetry. The equatorial Cl atoms of the anion display an umbrella effect with significant bending towards the tetrahydrofuran fragment, as observed before for similar anions. The cation exhibits bond lengths and angles in the expected range.

Comment

A single crystal of the title compound, (I), was isolated from a mixture of $Li_2[Et_2NB(C_5H_4)_2]$ and $[ZrCl_4(THF)_2]$ in toluene.



(I)

The structural parameters of (I) (Fig. 1) in the solid state resemble those of related compounds. In particular, the anion has been reported several times before (Scholz *et al.*, 1993; Polamo & Leskela, 1997; Alvanipour *et al.*, 1998; Bosch *et al.*, 1998; Faldt *et al.*, 2000). All bond lengths and angles are comparable to the reported ones and hence will not be discussed any further. The structural properties of the *N*,*N*-diethylpyrolidinium cation, however, have not been described in the literature before. Related structurally authenticated cations include the *N*-propyl-*N*-methylpyrolidinium ion (Golding *et al.*, 2001) and the spiro compound 5-azonia-spiro[4.4]nonane (Monkowius *et al.*, 2004). The N—C bond distances in the cation of the title compound range between 1.499 (4) and 1.511 (4) Å and hence are consistent with the bond lengths reported for the aforementioned species. The C—C distances lie in the expected range with values from 1.505 (6) up to 1.537 (6) Å. Since all atoms are sp^3-hybridized, bond angles of close to 109° are expected. Indeed, all angles between atoms constituting the heterocycle were found to be slightly smaller [102.2 (3) to 106.3 (3)°], whereas all corresponding angles between ring-atoms and their respective *exo*-substituents are more obtuse with values of 111.4 (2)–114.5 (3)°. Similar findings were made for the reference structures.

Experimental

Crystals of the title compound were isolated as a side product from the reaction between $Li_2[Et_2NB(C_5H_4)_2]$ (1.46 g, 6.5 mmol) and

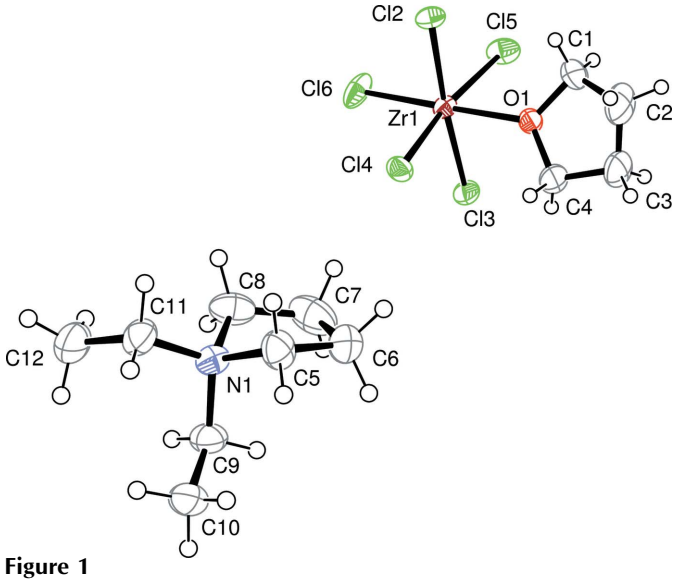


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

$[ZrCl_4(THF)_2]$ (2.45 g, 6.5 mmol). Suitable single crystals were grown from a toluene solution at 238 K over a period of three weeks (yield 0.36 g, 12%).

Crystal data

$(C_8H_{18}N)[ZrCl_5(C_4H_8O)]$	$Z = 8$
$M_r = 468.81$	$D_x = 1.581$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation
$a = 15.4148$ (17) Å	$\mu = 1.23$ mm^{-1}
$b = 14.9772$ (17) Å	$T = 173$ (2) K
$c = 17.0633$ (19) Å	Block, yellow
$V = 3939.4$ (8) Å^3	0.35 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer	54795 measured reflections
φ and ω scans	3922 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	3440 reflections with $I > 2\sigma(I)$
$T_{min} = 0.668$, $T_{max} = 0.841$	$R_{int} = 0.028$
	$\theta_{max} = 26.2°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 4.0936P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.034$	$(\Delta/\sigma)_{max} = 0.027$
$wR(F^2) = 0.108$	$\Delta\rho_{max} = 1.02$ e Å^{-3}
$S = 1.10$	$\Delta\rho_{min} = -0.52$ e Å^{-3}
3922 reflections	
183 parameters	
H-atom parameters constrained	

H atoms were placed at idealized positions and treated as riding atoms with C—H = 0.98 (CH_3) and 0.99 Å (CH_2); U_{iso}(H) values were fixed at 1.5 (for primary H atoms) and 1.2 times (secondary H atoms) U_{eq} of the attached C atom. The highest residual density peak is located 0.2 Å from atom Cl2.

Data collection: *SMART-NT* (Bruker, 1997); cell refinement: *SAINT-NT* (Bruker, 1997); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *XP* in *SHELXTL*.

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