The solid state structure and reactivity of NbCl₅'(N,N'-Dicyclohexylurea) in solution: Evidence for co-ordinated urea dehydration to the relevant carbodiimide

Michele Aresta,^{*^a} Angela Dibenedetto,^a Paolo Stufano,^a Brunella Maria Aresta,^b Sabino Maggi,^b Imre Pápai,^c and Tibos András Rokob^c

Supporting Information

Figure S1. The asymmetric cell unit of **1a**. The H atoms have an arbitrary dimension. Intramolecular H-bondings are represented with continuous black lines, while intermolecular H-bondings are represented by red dotted lines. Ellipsoids are drawn at 30 % probability. H-atoms are represented by spheres of arbitrary radius.

Figure S2. Representation of the unit cell of 1a.

Figure S3. 3D architecture of **1a** as determined by H-bonding of different intensity. Zig-zag motifs are clearly evident. One is generated by the C-H^{...}Cl bonds which produce a "zig-zag" motif along axis *a*. A second can be identified along the cell diagonal again generated by C-H^{...}Cl bonds integrated with weaker N-H^{...}Cl bonds. These two extended interactions generate the **2D** structure that is then repeated in the third direction with a packing along axis *b* in parallel plans due to C-H^{...}Cl and C-H^{...}N bonds to afford the **3D** architecture.

Table S1. D-H...A distances (Å) in the complex NbCl₅·DCU **1a** and the angles of the set of atoms



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Donor-H…Acceptor	D-H	H…Acceptor	Distance Donor… Acceptor	Angle (Donor- H…Acceptor)
$C(8A)-H(8A)-Cl(2B)^{(1)}$	0.980(7)	2.985(4)	3.855(9)	148.54 (36)
$N(1A)-H(1A)-Cl(2B)^{(1)}$	0.860(6)	2.975(3)	3.808(7)	163.78(46)
$C(9A)-H(9A2)-Cl(3B)^{(2)}$	0.970(11)	2.998(3)	3.876(12)	151.09(64)
$C(6A)-H(6A2)\cdots N(1B)^{(2)}$	0.970(12)	2.841(7)	3.735(14)	153.65(67)
$C(11A)-H(11A)-Cl(5B)^{(3)}$	0.970(8)	2.986(3)	3.764(8)	138.06(53)
$C(13A)-H(13B)-Cl(2B)^{(4)}$	0.970(11)	2.955(3)	3.877(12)	159.22(34)
C(8B)-H(8B)…Cl(2A) ⁽⁵⁾	0.980(4)	2.936(3)	3.812(9)	149.27(38)
$C(10B)-H(10D)-Cl(1A)^{(5)}$	0.970(13)	2.982(4)	3.813(14)	144.37(77)
N(1B)-H(1B)····Cl(2A) ⁽⁵⁾	0.860(6)	2.938(3)	3.770(7)	163.29(46)
$C(6B)-H(6B1)\cdots N(1A)^{(6)}$	0.970(10)	2.898(7)	3.784(12)	152.40(58)
C(13B)-H(13C)Cl(4A) ⁽⁶⁾	0.970(13)	2.988(3)	3.713(12)	132.58(69)
C(9B)-H(9D)····Cl(4A) ⁽⁷⁾	0.970(12)	2.997(4)	3.828(14)	144.39(75)
C(13B)-H(13D)···Cl(3A ⁽⁷⁾	0.970(14)	2.967(4)	3.776(16)	141.74(84)

Table S1. D-H...A distances (Å) in the complex NbCl₅·DCU **1a** and the angles (°) of the set of atoms with esd.

Symmetry code: (1) -x+1, +y-1/2, -z+2; (2) x+1,+y-1,+z; (3) -x+2,+y-1/2, -z+2; (4) x+1, +y, +z; (5) -x, +y+1/2, -z+1; (6) x-1, +y, +z; (7) -x+1, +y+1/2, -z+1; (6) x-1, +y, +z; (7) -x+1, +y+1/2, -z+1; (7) +x+1, +y+1/2, -z+