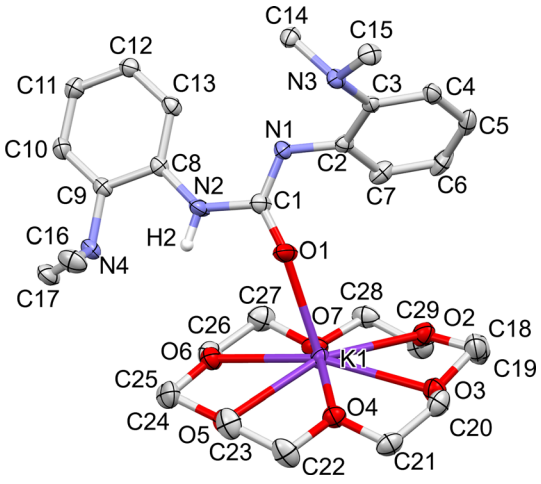
Dabeen Hong and Kyounghoon Lee*

Crystal structure of (1,4,7,10,13,16-hexaoxacyclooctadecane-κ^{6}O^6)-((*Z*)-*N*,*N*′-bis(2-(dimethylamino)phenyl)carbamimidato-κ^1*N*) potassium(I)



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Abstract

$C_{29}H_{45}KN_4O_7$, $P2_1/c$ (no. 14), a = 9.0016(2) Å, b = 23.5942(7) Å, c = 14.9186(4)Å, β = 102.509(1), V = 3093.28(14) Å^3, R_{gt} = 0.049, wR_{ref} = 1270, T = 100K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

1 Source of material

1,3-bis(2-(dimethylamino)phenyl)urea (20.8 mg, 0.070 mmol) was treated with KC_8 (9.5 mg, 0.070 mmol) and 18-crown-6 (18.5 mg, 0.070 mmol) in THF. The mixture was stirred overnight at room temperature, filtered through a pad of Celite, and evaporated to dryness under vacuum. It was recrystallized from the solution in THF by the vapor diffusion of pentane.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.34 × 0.18 × 0.13 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.22 mm^{-1}
Diffractometer, scan mode:	Bruker D8, φ and ω scans
θ_{max}, completeness:	27.5°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	66162, 7112, 0.072
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 6,137
$N(param)_{refined}$:	382
Programs:	Bruker[1], SHELX[2,3], Olex2[4], Mercury[5]

2 Experimental details

The structure was solved using the intrinsic phasing method SHELXT and refined using least square method SHELXL. The positional disorder of oxygen atom was modeled by refining over two positions each atom while setting the sum of the site occupancy factor 1. Hydrogen atoms on carbons were idealized using riding models, while hydrogen atoms on nitrogen were located from difference Fourier map inspection.

3 Comment

Urea compounds have been extensively used for chemical, pharmaceutical, and agrochemical industries due to their remarkable properties such as intra and intermolecular hydrogen bondings.[6–9] Aromatic-substituted urea compounds are also interesting classes of chemicals because of the extended π conjugations and flexibility. There is one title complex in the asymmetric unit.

A single proton was deprotonated from 1,3-bis(2-(dimethylamino)phenyl)urea to form a coordinated imidate. C–O bond distance of 1.257(2) Å is slightly elongated than the C–O

***Corresponding author: Kyounghoon Lee**, Department of Chemical Education and Research Institute of Natural Science, Gyeongsang National University, Gyeongnam 52828, Republic of Korea, E-mail: klee1@gnu.ac.kr. https://orcid.org/0009-0007-3503-5640
Dabeen Hong, Department of Science Education, Gyeongsang National University, Gyeongnam 52828, Republic of Korea

bond distance of 1.23 Å in diphenylurea.[10] The C1–N1 bond distance of 1.315(2) Å is slightly shorter than the C–N bond in diphenylurea, while the C1–N2 bond distance of 1.416(2) Å is slightly elongated.[10] The asymmetric environment also twisted the O–C–N angles of 114.12(15) and 129.27(17)°. The urea moieties and one aromatic ring were placed in the same plane, while the other aromatic ring is twisted with a torsion angle of 48.61(19)°. Intramolecular hydrogen bonding was found between the deprotonated nitrogen N1 and H13–C13. The donor-acceptor distance of 2.895(2) Å and donor-hydrogen-acceptor angle of 122.82(11)° indicated a weak hydrogen bonding interaction.

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