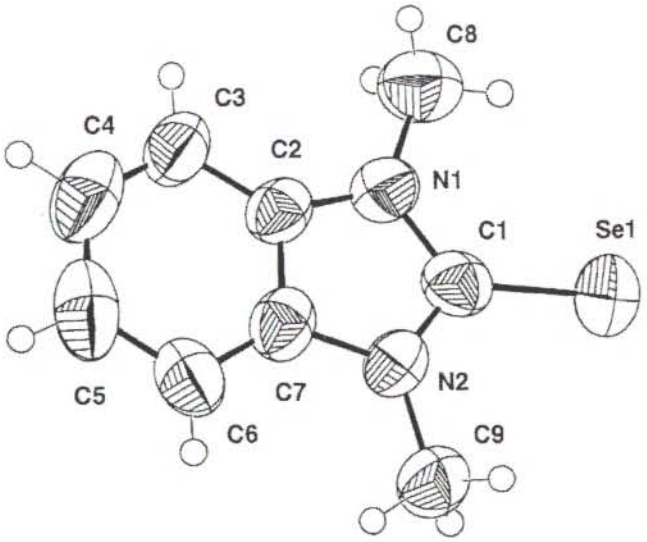
Z. Kristallogr. NCS 214 (1999) 295–296

Crystal structure of 1,3-dimethylbenzimidazole-2-selenone, $C_9H_{10}N_2Se$

A. Aydın*,[I], H. Soylu[I], H. Küçükbay[II], M. Akkurt[III] and F. Ercan[IV]

[I] Gazi University, Gazi Education Faculty, Department of Physics Education, 06500 Beşevler, Ankara, Turkey
[II] Inönü University, Faculty of Arts and Sciences, Department of Chemistry, 44069 Malatya, Turkey
[III] Erciyes University, Faculty of Arts and Sciences, Department of Physics, 38039 Kayseri, Turkey
[IV] Hacettepe University, Department of Physics Engineering, 06532 Beytepe, Ankara, Turkey

Received November 9, 1998, CCDC-No. 1267/57



Abstract

$C_9H_{10}N_2Se$, monoclinic, $P12_1/n1$ (No. 14), $a = 7.414(1)$ Å, $b = 14.339(1)$ Å, $c = 9.225(1)$ Å, $\beta = 107.70(1)°$, $V = 934.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.038$, $R_w(F^2) = 0.115$, $T = 293$ K.

Source of material

Selenium (0.56 g; 7.16 mmol) was added to a solution of bis(1,3-dimethylbenzimidazolidine-2-ylidene) (0.95 g; 3.25 mmol) in toluene (25 cm^3) and heated under reflux for 4 h. Then the solution was filtered while hot to remove excess of selenium. After cooling the solution to approx. 323 K pentane (15 cm^3) was added. Upon cooling the solution to 243 K white crystalls of the title compound were obtained (1.4 g; 96%, mp = 433 K – 434 K).

All reactions were performed under an argon or nitrogen atmosphere with use of schlenk techniques [1]. The solvents were deoxygenated and dried by standard methods [2].

Discussion

Electron-rich olefins are powerful reducing agents [3]. It is known that the ultimade oxidation product of electron-rich olefins [3-5] with air is urea; sulphur, selenium and tellurium react similarly to form the corresponding anologues [6, 7]. The oxidation rate is strongly dependent on the availability of electrons and hence on the substituents on the nitrogen. There are extensive studies about cyclic ureas having imidazolidine moiety including their X-ray crystal structure [8]. Less attention has been paid to the cyclic urea which contains benzimidazole moiety and there is no example known for the X-ray crystal structure determination. The aim of this study was to elucidate crystal structure of previously synthesised cyclic urea having benzimidazole moiety [9] and compare with imidazole analogues.

In the compound, N1–C1 and N2–C1 bond lengths resemble almost with N1–C1 and N2–C1 bond lengths in the bis(1-methyl-3-ethylbenzimidazolidine-2-ylium) tetrafluoroborate [10]. The N1–C1 bond length is 1.377(8) Å and N2–C1 bond length is 1.382(8) Å.

Table 1. Data collection and handling.

Crystal:	cream, prismatic, size 0.35 x 0.40 x 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	39.64 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	45.38°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1157, 1157
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1026
$N(param)_{refined}$:	110
Programs:	SHELXS-86 [11], SHELXL-97 [12], ORTEPII [13]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.1576	0.152	1.2302	0.092
H(4)	4e	0.2295	0.0226	1.369	0.114
H(5)	4e	0.3157	–0.1175	1.2765	0.117
H(6)	4e	0.3176	–0.1237	1.0178	0.095
H(8A)	4e	0.0854	0.2875	0.8401	0.133
H(8B)	4e	0.1888	0.2899	1.0157	0.133
H(8C)	4e	–0.022	0.2561	0.9543	0.133
H(9A)	4e	0.2934	–0.0941	0.773	0.117
H(9B)	4e	0.3509	–0.0162	0.6763	0.117
H(9C)	4e	0.1376	–0.0443	0.6419	0.117

* Correspondence author (e-mail: bgunes@sirius.gazi.edu.tr)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Se(1)	4*e*	0.1494(1)	0.18246(5)	0.61588(8)	0.109(1)	0.089(1)	0.068(1)	–0.0027(3)	0.0302(6)	0.0128(3)
N(1)	4*e*	0.1634(8)	0.1610(4)	0.9286(7)	0.078(3)	0.068(3)	0.068(4)	–0.005(3)	0.030(3)	–0.009(3)
N(2)	4*e*	0.2334(7)	0.0346(3)	0.8287(5)	0.069(3)	0.071(3)	0.048(3)	–0.001(2)	0.013(2)	–0.002(2)
C(1)	4*e*	0.1830(8)	0.1230(5)	0.7971(7)	0.061(3)	0.063(4)	0.076(4)	–0.014(3)	0.022(3)	–0.008(3)
C(2)	4*e*	0.1993(8)	0.0968(4)	1.0414(7)	0.058(3)	0.073(4)	0.054(4)	–0.007(3)	0.015(3)	–0.007(3)
C(3)	4*e*	0.192(1)	0.0981(6)	1.1888(8)	0.083(4)	0.094(5)	0.054(4)	–0.013(4)	0.023(3)	–0.006(4)
C(4)	4*e*	0.233(1)	0.0210(8)	1.269(1)	0.079(5)	0.146(8)	0.059(5)	–0.019(5)	0.018(3)	–0.002(5)
C(5)	4*e*	0.285(1)	–0.0654(6)	1.214(1)	0.083(5)	0.124(7)	0.081(5)	–0.015(4)	0.016(4)	0.037(5)
C(6)	4*e*	0.288(1)	–0.0694(5)	1.0606(9)	0.080(4)	0.070(4)	0.080(5)	–0.008(3)	0.015(4)	0.013(4)
C(7)	4*e*	0.2431(8)	0.0131(5)	0.9794(7)	0.061(3)	0.087(4)	0.062(4)	–0.012(3)	0.023(3)	–0.002(3)
C(8)	4*e*	0.098(1)	0.2569(6)	0.9352(9)	0.092(5)	0.078(5)	0.099(5)	–0.001(4)	0.034(4)	–0.014(4)
C(9)	4*e*	0.256(1)	–0.0361(5)	0.7206(9)	0.091(5)	0.076(4)	0.075(5)	0.009(3)	0.036(4)	–0.003(4)

Acknowledgments. The authors wish to acknowledge the purchase of CAD4 diffractometer under. Grand DPT/TBAG1 of The Scientific and Technical Research Concil of Turkey.

References

1. Shriver, D. F.; Drezdzon, M. A.: The Manipulation of Air Sensitive Compounds. John Wiley & Sons., New York 1986.
2. Perrin, D. D.; Armerego, W. L. F.; Perrin, D. R. M.: Purfication of Laboratory Chemicals. 2nd. Ed., Pergammon Press 1986.
3. Lappert, M. F.: Characterisation and crystal structure. J. Organomet. Chem. **358** (1988) 185-185.
4. Wiberg, N.: Tetraaminoethylenes as strong electron donors. Angew. Chem. (Int. Edn. Engl.) **7** (1968) 766-779.
5. Winberg, H. E.; Downing, J. R.; Coffman, D. D.; Carnahan, J. E.; Brown, M.: Tetraaminoethylenes. J. Am. Chem. Soc. **87** (1968) 2055-2057.
6. Roterding, F.; Scheeren, J. W.; Laarhoven, W. H.: Controlled chemilumiescence during the oxidation of tetraaryl-$\Delta^{2,2'}$-biimidazolidines by triplet oxygen in the presence of fluorecers. Tetrahedron Lett. **24** (1983) 2307-2310.
7. Lappert, M. F.; Martin, T. R.; McLaughlin, G. M.: Telluroureas and derived transition metal complexes: the crystal and molecular structure of $[Cr(CO)_5\{Te{=}CN(Et)CH_2CH_2NEt\}]$. J. Chem. Soc. Chem. Commun. (1980) 635-637.
8. Jasim, H. A.: Synthesis and characterisation of unusaul tetraaminoalkenes (enetetramines). D. Phil. Thesis, University of Sussex, Great Britain 1986.
9. Çetinkaya, B.; Çetinkaya, E.; Küçükbay, H.; Durmaz, R.: Synthesis and antimicrobial activity od electron-rich olefine derived cyclic ureas. Arzneim.-Forsch./Drug Res. **46**(II) (1996) 1154-1158.
10. Aydin, A.; Soylu, H.; Günes, B.; Akkurt, M.; Ercan, F.; Küçükbay, H.; Çetinkaya, E.: Preparation and X-ray structure of bis(1-methyl-3-ethylbenzimidazolidine-2-ylium) tetrafluoroborate. Z. Kristallogr. **213** (1998) 473-476.
11. Sheldrick, G. M.: SHELXS-86. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1990.
12. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
13. Johnson, C. K.: ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA 1976.