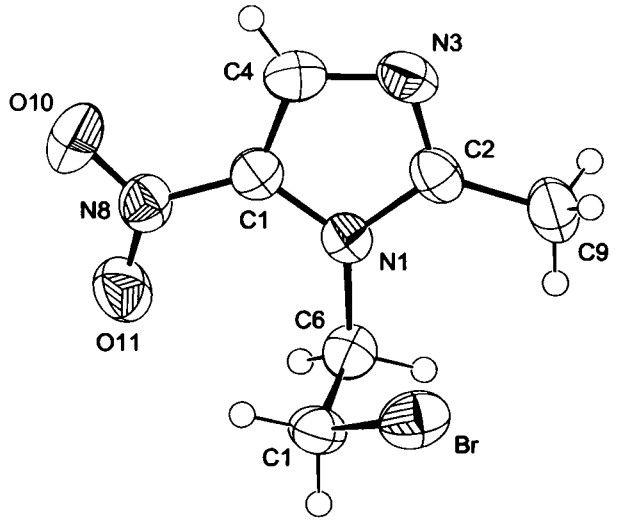
Z. Kristallogr. NCS 221 (2006) 49-50

Crystal structure of 1-(2-bromoethyl)-2-methyl-5-nitroimidazole, $C_6H_8BrN_3O_2$

T. L. Balliano*, M. A. Pereira, C. A. De Simone, V. R. S. Malta, N. Velasquez, A. G. Cioletti and M. O. F. Goulart

Universidade Federal de Alagoas, Departamento de Química, 57072-970 - Maceió, AL, Brazil

Received December 22, 2005, accepted and available on-line March 2, 2006; CCDC no. 1267/1706



Abstract

$C_6H_8BrN_3O_2$, monoclinic, $P12_1/a1$ (no. 14), a = 6.3952(2) Å, b = 11.1267(3) Å, c = 12.0785(4) Å, β = 97.274(2)°, V = 852.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.128, T = 293 K.

Source of material

Mesilatemetronidazole and bromidemetronidazole were prepared as described in the literature [1]. To a dimethylformamide solution (30 ml) of mesilatemetronidazole (453.6 mg, 1.82 mmol) was added NaBr (1872.5 mg, 18.18 mmol). This mixture was stirred for 5 h at 353 K. The solvents were removed and the residue recrystallized from ether. Crystals were collected and washed with diethyl ether added in presence of $NaSO_4$. Large crystals suitable for X-ray crystal structure determination were obtained after filtration and drying at room temperature (yield 81 %).

Discussion

Nitroimidazole derivatives are extensively used in the treatment of anaerobic infections and more recently as anti-*Helicobacter pylori* agents. They are largely studied by electrochemical methods due to the great importance of the redox potential for the understanding of the mechanism of biological activity [2]. The title compound shows significant activity against metronidazole-resistant *Helicobacter pylori* [3].

In the crystal structure, the imidazole ring is a well defined plane with an average deviation of 0.006 Å. The nitro N atom lies 0.057(3) Å below the plane. The two other groups attached to the ring are located on the opposite side of the plane, with deviations of C9 and C5 atoms from the best plane of –0.011(4) Å and 0.102(3) Å, respectively. 1-(2-hydroxyethyl)-2-methyl-5-nitroimidazole (metronidazole) [4] crystallizes non-isotypically because of the stronger intermolecular O–H···O bonds and only weaker C–H···Br interactions formed in the title structure (apart from N–O···H–C interactions appearing in both structures).

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.2 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	47.85 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9141, 1947
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1669
$N(param)_{refined}$:	109
Programs:	SHELXS-97 [5], SHELXL-97 [6], ORTEP-3 [7], WinGX [8]

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

Atom	Site	x	y	z	U_{iso}
H(5A)	4e	–0.2577	0.1546	0.2220	0.051
H(5B)	4e	–0.3435	0.0801	0.1157	0.051
H(6A)	4e	–0.0057	0.1480	0.0976	0.056
H(6B)	4e	–0.0042	0.0072	0.1027	0.056
H(2)	4e	–0.3258	–0.2880	0.3233	0.060
H(4A)	4e	–0.2413	0.1601	0.3869	0.087
H(4B)	4e	–0.1334	0.0860	0.4888	0.087
H(4C)	4e	–0.3791	0.1012	0.4707	0.087

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}
Br	4e	0.19721(6)	0.08503(4)	0.27178(3)	0.0435(3)	0.0643(3)	0.0703(3)	–0.0084(1)	–0.0028(2)	0.0086(2)
N(1)	4e	–0.2759(4)	–0.0219(2)	0.2521(2)	0.028(1)	0.045(1)	0.037(1)	0.0016(9)	0.0036(9)	–0.0002(9)
C(6)	4e	–0.2394(5)	0.0809(2)	0.1815(3)	0.044(2)	0.040(2)	0.042(2)	0.006(1)	–0.003(1)	0.004(1)
C(5)	4e	–0.2998(5)	–0.1411(3)	0.2234(3)	0.031(1)	0.047(2)	0.042(2)	–0.002(1)	0.002(1)	–0.001(1)

* Correspondence author (e-mail: tlb@qui.ufal.br)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	–0.0210(6)	0.0795(3)	0.1456(3)	0.049(2)	0.051(2)	0.040(2)	–0.006(1)	0.006(1)	0.008(1)
N(8)	4*e*	–0.3174(4)	–0.1877(3)	0.1140(2)	0.040(1)	0.057(2)	0.045(1)	–0.007(1)	0.000(1)	–0.008(1)
C(4)	4*e*	–0.3112(5)	–0.2050(3)	0.3191(3)	0.044(2)	0.050(2)	0.055(2)	–0.005(1)	0.006(1)	0.008(2)
O(10)	4*e*	–0.3260(6)	–0.2978(3)	0.1044(3)	0.091(2)	0.053(2)	0.076(2)	–0.016(1)	0.004(2)	–0.020(1)
O(11)	4*e*	–0.3259(5)	–0.1190(3)	0.0340(2)	0.064(2)	0.077(2)	0.042(1)	–0.007(1)	–0.001(1)	–0.004(1)
C(2)	4*e*	–0.2749(5)	–0.0189(3)	0.3652(2)	0.033(1)	0.060(2)	0.038(2)	0.001(1)	0.006(1)	–0.002(1)
N(3)	4*e*	–0.2977(5)	–0.1280(3)	0.4071(2)	0.046(2)	0.065(2)	0.044(1)	–0.007(1)	0.011(1)	0.006(1)
C(9)	4*e*	–0.2554(7)	0.0919(3)	0.4339(3)	0.063(2)	0.067(2)	0.045(2)	–0.001(2)	0.011(2)	–0.013(2)

Acknowledgments. This work has received partial support from CNPq, CAPES, FAPEAL, and FINEP.

References

1. Kajfez, F.; Sunjic, V.; Kolbah, D.; Fajdiga, T.; Oklobdzija, M.: 1-substitution in 2-methyl-4(5)-nitroimidazole, synthesis of compounds with potential antitrichomonal activity. J. Med. Chem. **11** (1968) 167-170.
2. Cavalcanti, J. C. M.; de Abreu, F. C.; Oliveira, N. V.; de Moura, M. A. B. F.; Chaves, J. G.; Alves, R. J.; Bertinaria, M.; Fruttero, R.; Goulart, M. O. F.: Effect of the leaving group on the electrodic reduction mechanism of anti-*Helicobacter pylori* metronidazole derivatives, in aprotic and protic media. Bioelectrochemistry **63** (2004) 353-357.
3. Alves, R. J.: Personal communication (2005).
4. Blaton, N. M.; Peeters, O. M.; De Ranter, C. J.: 2-(2-Methyl-5-nitro-1-imidazolyl)ethanol (Metronidazole). Acta Crystallogr. **B35** (1979) 2465-2467.
5. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
6. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
7. Farrugia, L. J.: ORTEP-3 for Windows – a version of ORTEP-III with a Graphical User Interface (GUI). J. Appl. Crystallogr. **30** (1997) 565.
8. Farrugia, L. J.: WinGX suite for small-molecule single-crystal crystallography. J. Appl. Crystallogr. **32** (1999) 837-838.