Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[2-(5-Nitro-1*H*-benzimidazol-1-yl)ethyl]-morpholinium chloride

Mehmet Akkurt,[a]* Sevim Türktekin,[a] Hasan Küçükbay,[b] Ülkü Yılmaz[b] and Orhan Büyükgüngör[c]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Chemistry, Faculty of Arts and Sciences, Ínönü University, 44280 Malatya, Turkey, and [c]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey

Correspondence e-mail: akkurt@erciyes.edu.tr

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
R factor = 0.027
wR factor = 0.069
Data-to-parameter ratio = 14.3

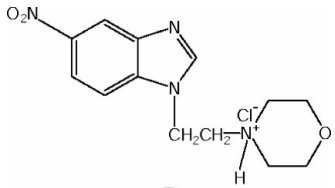
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 December 2004
Accepted 15 December 2004
Online 24 December 2004

The title compound, $C_{13}H_{17}N_4O^+\cdot Cl^-$, was synthesized from 1-(2-methoxyethyl)-5-nitrobenzimidazole and *N*-(2-chloroethyl)morpholine hydrochloride in dimethylformamide. The crystal structure has been determined at 100 K and exhibits an intramolecular N—H···Cl, and intermolecular C—H···Cl and C—H···O interactions.

Comment

Benzimidazole derivatives occupy an important place among synthetic medical preparations due to the wide spectrum of their pharmacological activity (Kataev *et al.*, 2002). Nitroaromatic compounds such as nitrobenzenes, nitrofurans, nitroimidazaoles and nitrobenzimidazoles are widely used as pharmaceuticals, food additives and explosives. For manifestation of their therapeutic and/or cytotoxic properties, most nitroaromatics should undergo single- or two-electron enzymatic reduction in organisms. Nitrobenzimidazoles act as relatively efficient substrates for rat DT-diaphorase (Sarlauskas *et al.*, 1997). We have synthesized and investigated the crystal structures of many benzimidazole derivatives which constitute an important class of heterocyclic compounds (Akkurt *et al.*, 2003; Akkurt, Öztürk, Şireci *et al.*, 2004; Akkurt *et al.*, 2004*a*,*b*; Akkurt, Öztürk, Küçükbay, Yılmaz *et al.*, 2004; Öztürk *et al.*, 2001, 2003, Türktekin, Akkurt, Orhan *et al.*, 2004; Türktekin, Akkurt, Şireci *et al.*, 2004). We also observed that many benzimidazole derivatives and related heterocyclic compounds have shown considerable antimicrobial activities against standard strains: *Enterococcus faecalis* (ATCC 29212), *Staphylococcus aureus* (ATCC 29213), *Escherichia coli* (ATCC 25922), *Pseudomonas aeruginosa* (ATCC 27853) and yeasts *Candida albicans* and *Candida tropicalis* (Küçükbay *et al.*, 2001, 2003, 2004). The aim of this study was to synthesize and elucidate the crystal structure of a new 5-nitrobenzimidazole compound, (I), containing a morpholine moiety.



(I)

The molecular geometry of (I) and the atomic numbering scheme are shown in Fig. 1. Selected geometric parameters are listed in Table 1. All geometric parameters are comparable with the results obtained from our previous studies on related benzimidazole derivatives (Akkurt, Öztürk, Şireci *et al.*, 2004; Akkurt *et al.*, 2004*a*,*b*; Akkurt, Öztürk, Küçükbay, Yılmaz *et al.*, 2004; Türktekin, Akkurt, Şireci *et al.*, 2004).

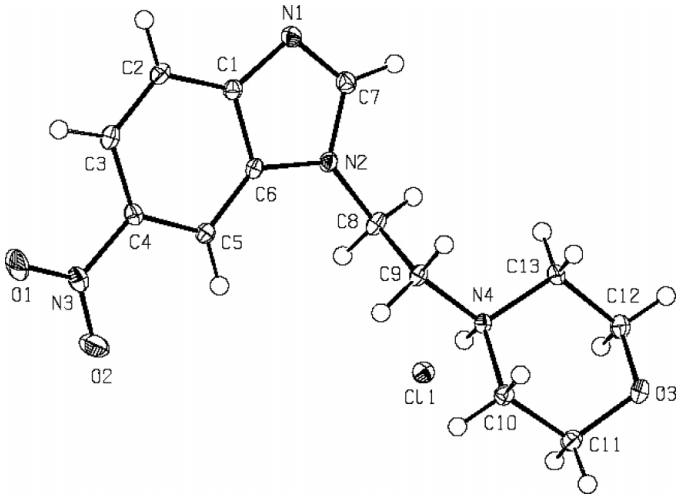


Figure 1
An *ORTEP3* (Farrugia, 1997) plot of the title compound, with the atom-numbering scheme and 50% probability displacement ellipsoids

In (I), the benzimidazole ring system (N2/C7/C8/C9/C10/C11/C12/N3/C13) is essentially planar and the maximum deviations from planarity are 0.016 (1) and −0.018 (1) Å for atoms C4 and C7, respectively. The conformation of the morpholine ring (O3/C11/C10/N4/C13/C12) is that of a chair, with puckering parameters Q_T = 0.586 (1) Å, θ = 3.4 (1)° and φ = 338 (2)° (Cremer & Pople, 1975).

Details of the hydrogen-bonding geometry are listed in Table 2 and shown in Fig. 2.

Experimental

The title compound was synthesized by nucleophilic substitution of 1-(2-methoxyethyl)-5-nitrobenzimidazole with *N*-(2-chloroethyl)morpholine hydrochloride. 1-(2-Methoxyethyl)-5-nitrobenzimidazole was synthesized from 2-methoxyethyl chloride and 5-nitrobenzimidazolium nitrate as indicated in the literature procedure of Küçükbay *et al.* (2001). A mixture of 1-(2-methoxyethyl)-5-nitrobenzimidazole (2.00 g; 9.05 mmol) and *N*-(2-chloroethyl)morpholine hydrochloride (1.85 g; 9.95 mmol) in dimethylformamide (8 ml) was heated on a water bath for 3 h. All volatiles were then removed *in vacuo*. The crude product obtained was crystallized from EtOH/Et_2O (3:1) mixture (yield: 1.90 g, 67%; m.p. 556–557 K). ^{1}H NMR (D_2O): δ, 3.67 (*t*, CH_2CH_2-morpholine, 2 H), 3.63 (*t*, ring methylene, 4 H), 3.86 (*t*, CH_2CH_2-morpholine, 2 H), 4.71 (*t*, ring methylene, 4 H), 7.56–8.36 (*m*, Ar—H, 4 H). ^{13}C NMR (D_2O): δ 39.34, 52.09, 54.70, 63,62, 107.53, 118.73, 119.41, 143.68. Analysis calculated for $C_{13}H_{17}ClN_4O_3$: C 49.92, H 5.44, N 17.92%; found: C 49.87, H 5.44, N 17.76%.

Crystal data

$C_{13}H_{17}N_4O^+\cdot Cl^-$
M_r = 312.76
Triclinic, $P\overline{1}$
a = 7.0522 (5) Å
b = 9.9537 (8) Å
c = 9.9895 (7) Å
α = 83.607 (6)°
β = 80.386 (6)°
γ = 89.477 (6)°
V = 687.04 (9) Å^3
Z = 2
D_x = 1.512 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 23590 reflections
θ = 2.8–29.3°
μ = 0.30 mm^{-1}
T = 100 K
Prism, colorless
0.23 × 0.22 × 0.20 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED*32; Stoe & Cie, 2002)
T_{min} = 0.935, T_{max}= 0.943
14 617 measured reflections
3692 independent reflections
3381 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 29.1°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.069
S = 1.06
3692 reflections
258 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2 + 0.269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N3	1.2265 (13)	N2—C7	1.3657 (13)
O2—N3	1.2222 (13)	N2—C8	1.4543 (13)
O3—C11	1.4237 (12)	N3—C4	1.4601 (13)
O3—C12	1.4250 (13)	N4—C9	1.5002 (12)
N1—C1	1.3826 (13)	N4—C10	1.5007 (13)
N1—C7	1.3102 (13)	N4—C13	1.4985 (13)
N2—C6	1.3844 (12)		
C11—O3—C12	109.12 (8)	N1—C1—C6	110.52 (8)
C1—N1—C7	104.00 (8)	N3—C4—C5	117.62 (9)
C6—N2—C7	105.88 (8)	N3—C4—C3	117.70 (8)
C6—N2—C8	128.79 (8)	N2—C6—C1	104.95 (8)
C7—N2—C8	125.08 (8)	N2—C6—C5	132.20 (9)
O1—N3—O2	123.14 (9)	N1—C7—N2	114.65 (9)
O1—N3—C4	118.49 (9)	N2—C8—C9	111.71 (8)
O2—N3—C4	118.36 (9)	N4—C9—C8	110.25 (8)
C9—N4—C10	111.11 (7)	N4—C10—C11	110.06 (8)
C9—N4—C13	111.83 (7)	O3—C11—C10	111.16 (8)
C10—N4—C13	109.43 (7)	O3—C12—C13	110.70 (8)
N1—C1—C2	129.05 (9)	N4—C13—C12	109.55 (8)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···Cl1	0.903 (14)	2.151 (14)	3.0517 (9)	176.0 (12)
C3—H3···O3^{i}	0.963 (16)	2.399 (16)	3.2840 (13)	152.6 (13)
C5—H5···Cl1^{ii}	0.937 (15)	2.760 (15)	3.6627 (10)	162.1 (12)
C7—H7···O1^{iii}	0.961 (15)	2.334 (15)	3.2845 (13)	169.9 (12)
C9—H9*A*···Cl1^{ii}	0.963 (14)	2.652 (14)	3.5562 (10)	156.6 (11)
C12—H12*B*···O2^{ii}	0.979 (14)	2.519 (14)	3.3412 (14)	141.5 (11)
C13—H13*A*···O2^{iii}	0.966 (14)	2.425 (14)	3.2462 (13)	142.6 (11)

Symmetry codes: (i) $x, y-1, z+1$; (ii) $-x+1, -y+1, -z$; (iii) $x, y+1, z$.

All H atoms were found in difference Fourier maps and refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant F.279 of the University Research Fund). HK and ÜY also thank İnönü

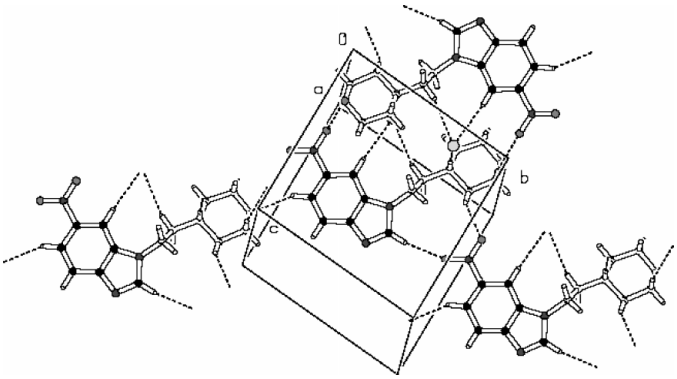


Figure 2
View of the hydrogen bonding (shown as dashed lines) of (I).

University Scientific Research Unit (BAPB-2002/06 directed project) for financial support for this study.

References

Akkurt, M., Öztürk, S., Küçükbay, H., Okuyucu, N. & Fun, H.-K. (2003). *Acta Cryst.* E**59**, o786–o788.
Akkurt, M., Öztürk, S., Küçükbay, H., Orhan, E. & Büyükgüngör, O. (2004*a*). *Acta Cryst.* E**60**, o219–o221.
Akkurt, M., Öztürk, S., Küçükbay, H., Orhan, E. & Büyükgüngör, O. (2004*b*). *Acta Cryst.* E**60**, o219–o221.
Akkurt, M., Öztürk, S., Küçükbay, H., Yılmaz, Ü. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o2135–o2137.
Akkurt, M., Öztürk, S., Şireci, N., Küçükbay, H. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o1185–o1187.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kataev, V. A., Spirikhin, L. V., Khaliullin, A. N. & Gailyunas, I. A. (2002). *Russ. J. Org. Chem.* **38**, 1057–1059.
Küçükbay, H., Durmaz, R., Güven, M. & Günal, S. (2001). *Arzneim. Forsch./ Drug Res.* **51**, 420–424.
Küçükbay, H., Durmaz, R., Okuyucu, N., Günal, S. & Kazaz, C. (2004). *Arzneim. Forsch./Drug Res.* **54**, 64–68.
Küçükbay, H., Durmaz, R., Orhan, E. & Günal, S. (2003). *Il Farmaco*, **58**, 431–437.
Öztürk, S., Akkurt, M., Küçükbay, H. & Fun, H.-K. (2001). *Anal. Sci.* **17**, 1015–1016.
Öztürk, S., Akkurt, M., Küçükbay, H., Okuyucu, N. & Fun, H.-K. (2003). *Acta Cryst.* E**59**, o1014–o1016.
Sarlauskas, J., Dickancaite, E., Nemeikaite, A., Anusevicius, Z., Nivinskas, H., Segura-Aguilar, J. & Cenas, N. (1997). *Arch. Biochem. Biophys.* **346**, 219–229.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
Türktekin, S., Akkurt, M., Orhan. E., Küçükbay, F. Z., Küçükbay, H. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, m1220–m1222.
Türktekin, S., Akkurt, M., Şireci, N., Küçükbay, H. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o817–o819.

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[2-(5-Nitro-1*H*-benzimidazol-1-yl)ethyl]-morpholinium chloride. Corrigendum

Mehmet Akkurt,[a]* Sevim Türktekin,[a] Hasan Küçükbay,[b] Ülkü Yılmaz[b] and Orhan Büyükgüngör[c]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Chemistry, Faculty of Arts and Sciences, Ínönü University, 44280 Malatya, Turkey, and [c]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey

Correspondence e-mail: akkurt@erciyes.edu.tr

In the paper by Akkurt, Türktekin, Küçükbay, Yılmaz & Büyükgüngör [*Acta Cryst.* (2005), E**61**, o166–o168], the experimental section is incorrect. The correct experimental section is given below.

Experimental

The title compound was synthesized by nucleophilic substitution of 5-nitrobenzimidazole with *N*-(2-chloroethyl)morpholine hydrochloride. A mixture of 5-nitrobenzimidazole (2.00 g, 12.27 mmol) and *N*-(2-chloroethyl)morpholine hydrochloride (2.28 g, 12.27 mmol) in DMF (8 ml) was heated on a water bath for 3 h. All volatiles were then removed *in vacuo*. The crude product obtained was crystallized from an EtOH/Et_2O (3:1) mixture (yield: 2.76 g, 72%; m.p. 556–557 K). ^{1}H NMR (D_2O): δ 3.67 (*t*, CH_2CH_2-morpholine, 2H), 3.63 (*t*, ring methylene, 4H), 3.86 (*t*, CH_2CH_2-morpholine, 2H), 4.71 (*t*, ring methylene, 4H), 7.56–8.36 (*m*, Ar-*H*, 4H). ^{13}C NMR (D_2O): δ 39.34, 52.09, 54.70, 63,62, 107.53, 118.73, 119.41, 143.68. Analysis calculated for $C_{13}H_{17}ClN_4O_3$: C 49.92, H 5.44, N 17.92%; found: C 49.87, H 5.44, N 17.76%.