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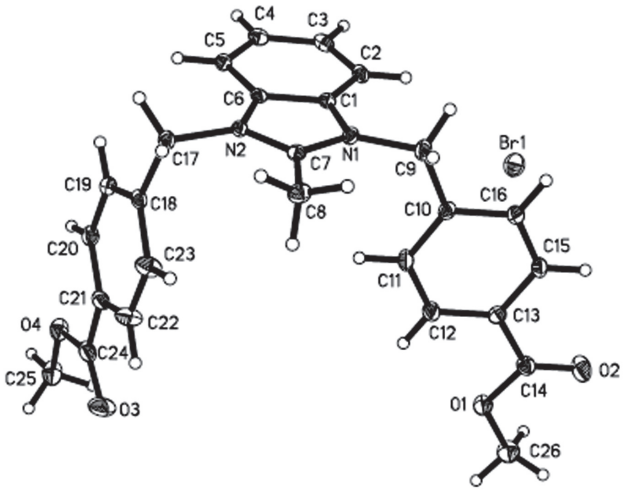
The crystal structure of 1,3-bis(4-(methoxycarbonyl)benzyl)-2-methyl-1*H*-benzo[*d*]imidazol-3-ium bromide, $C_{26}H_{25}BrN_2O_4$



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Abstract

$C_{26}H_{25}BrN_2O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 9.4202(5)$ Å, $b = 9.7871(5)$ Å, $c = 15.0135(9)$ Å, $\alpha = 106.775(3)°$, $\beta = 92.534(3)°$, $\gamma = 117.424(3)°$, $V = 1150.52(11)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0316$, $wR_{ref}(F^2) = 0.0671$, $T = 150(2)$ K.

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The title salt structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents were purchased and used as received. The title compound was synthesized according to reference [5]: Under stirring, 1.32 g 2-methyl-1*H*-benzo[*d*]imidazole

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Table 1: Data collection and handling.

Crystal:	Colorless needle
Size:	0.25 × 0.20 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.82 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22877, 4722, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3913
$N(param)_{refined}$:	302
Programs:	Bruker [1], SHELX [2], Olex2 [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.39309(3)	0.71322(3)	0.02283(2)	0.02496(8)
C1	0.1072(3)	0.1218(2)	0.12137(14)	0.0187(4)
C2	0.1948(3)	0.0379(3)	0.10799(15)	0.0235(5)
H2	0.306767	0.089780	0.104177	0.028*
C3	0.1099(3)	−0.1247(3)	0.10059(15)	0.0257(5)
H3	0.165067	−0.186495	0.091939	0.031*
C4	−0.0551(3)	−0.2009(3)	0.10548(15)	0.0243(5)
H4	−0.109411	−0.313483	0.099410	0.029*
C5	−0.1419(3)	−0.1170(2)	0.11894(15)	0.0205(4)
H5	−0.254238	−0.169101	0.121993	0.025*
C6	−0.0560(2)	0.0470(2)	0.12769(14)	0.0174(4)
C7	0.0236(3)	0.3049(3)	0.13856(14)	0.0203(4)
C8	0.0222(3)	0.4589(3)	0.14653(16)	0.0274(5)
H8A	0.046283	0.525499	0.213818	0.041*
H8B	−0.085767	0.432281	0.115859	0.041*
H8C	0.105323	0.520666	0.115185	0.041*
C9	0.3156(3)	0.3995(3)	0.12139(16)	0.0261(5)
H9A	0.307142	0.482360	0.099641	0.031*
H9B	0.354978	0.340718	0.072769	0.031*
C10	0.4394(3)	0.4866(2)	0.21385(15)	0.0210(5)
C11	0.3998(3)	0.5025(3)	0.30261(16)	0.0281(5)
H11	0.288188	0.453987	0.307343	0.034*
C12	0.5210(3)	0.5885(3)	0.38467(16)	0.0274(5)
H12	0.492320	0.598189	0.445161	0.033*
C13	0.6839(3)	0.6601(3)	0.37835(15)	0.0223(5)
C14	0.8211(3)	0.7525(3)	0.46280(16)	0.0272(5)
C15	0.7243(3)	0.6456(3)	0.28950(16)	0.0261(5)
H15	0.835935	0.695530	0.284935	0.031*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C16	0.6036(3)	0.5593(3)	0.20780(16)	0.0250(5)
H16	0.632508	0.549431	0.147356	0.030*
C17	−0.2665(3)	0.1412(3)	0.15632(15)	0.0218(5)
H17A	−0.352812	0.029976	0.115132	0.026*
H17B	−0.287221	0.222485	0.140107	0.026*
C18	−0.2724(3)	0.1611(3)	0.25980(15)	0.0210(5)
C19	−0.3324(3)	0.0252(3)	0.28832(16)	0.0246(5)
H19	−0.378555	−0.081781	0.241973	0.030*
C20	−0.3254(3)	0.0442(3)	0.38340(16)	0.0262(5)
H20	−0.365377	−0.049470	0.402176	0.031*
C21	−0.2600(3)	0.2003(3)	0.45202(16)	0.0259(5)
C22	−0.2056(3)	0.3353(3)	0.42323(18)	0.0358(6)
H22	−0.164047	0.441865	0.469264	0.043*
C23	−0.2114(3)	0.3160(3)	0.32841(17)	0.0335(6)
H23	−0.173291	0.409586	0.309610	0.040*
C24	−0.2381(3)	0.2271(3)	0.55538(17)	0.0306(5)
C25	−0.2742(3)	0.1033(4)	0.67171(18)	0.0413(7)
H25A	−0.156434	0.152491	0.694777	0.062*
H25B	−0.331708	−0.005105	0.677915	0.062*
H25C	−0.313128	0.174084	0.709422	0.062*
C26	0.9006(3)	0.8440(3)	0.62869(17)	0.0407(7)
H26A	0.959964	0.783925	0.628464	0.061*
H26B	0.853934	0.853249	0.685750	0.061*
H26C	0.975971	0.954040	0.628629	0.061*
N1	0.1527(2)	0.2819(2)	0.12819(12)	0.0199(4)
N2	−0.1038(2)	0.1657(2)	0.13919(12)	0.0173(4)
O1	0.77036(19)	0.7564(2)	0.54478(11)	0.0319(4)
O2	0.9624(2)	0.8150(3)	0.45785(13)	0.0473(5)
O3	−0.1664(2)	0.3611(2)	0.61727(12)	0.0447(5)
O4	−0.3057(2)	0.0864(2)	0.57299(11)	0.0331(4)

(10.0 mmol) was added to 50 mL acetone, then 4.56 g 4-(methoxycarbonyl) benzyl bromide (20.0 mmol) was added in portions. The mixture was stirred under reflux for 24 h. The volume was reduced to ca. 10 mL by rotary evaporation and then cooled to room temperature, after which a white precipitate appeared, washed with acetone, and air-dried, yield 72.6%. The precipitate was transferred to 20 mL of THF solution, then filtered after stirred for 5 min. The filtrate let evaporation for several hours to get colorless crystals yield 87.4%.

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropically ($U_{iso}(H) = 1.2U_{eq}(C)$ using a riding model with C—H = 0.95, 0.98 and 0.99 Å.

Comment

N-heterocyclic carbenes (NHCs) and metal NHCs have been focused on scientific researchers for catalysts and medicinal applications [6]. Known as important precusors for NHCs, 1,3-bis(4-(methoxycarbonyl)benzyl)-1*H*-imidazol-3-ium bromide and 1,3-bis(4-(methoxycarbonyl)benzyl)-1*H*-benzo[*d*]imidazol-3-ium bromide, or their derivatives, including metal NHCs, have been reported [7, 8]. Furthermore, lanthanide complexes have been studied [9].

The title organic salt as shown in the figure, the asymmetric unit consists of one 1,3-bis(4-(methoxycarbonyl)benzyl)-2-methyl-1*H*-benzo[*d*]imidazol-3-ium cation and one bromide anion. The skeleton of the benzimidazole is nearly planar. The two 4-(methoxycarbonyl)benzyl groups look like two arms, which adapt a configuration that the angles of C10···C9···N1 and C18···C17···N2 are 114° and 110°, respectively. There are three kinds of weak intermolecular short contacts, such as C—H···O, C—H···Br and C—H···π, which are linked each other to generate a three-dimensional structure. All bond lengths and angles are in normal ranges [5, 7–10].

References

1. Bruker. SAINT v8.37A. Bruker AXS Inc, Madison, WI, USA (2015).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Bourhis, L. J.; Dolomanov, O. V.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: The anatomy of a comprehensive constrained, restrained refinement program for the modern computing environment – Olex2 dissected. Acta Crystallogr. **A71** (2015) 59–75.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Crystallogr. **42** (2009) 339–341.
5. Huang, H.-R.; Guo, W.-J.; Du, Z.-Y.; Fang, Y.-X.; Zhang, K.: 1,3-Bis[4-(methoxycarbonyl)benzyl]benzimidazolium bromide monohydrate. Acta Crystallogr. **E66** (2010) o3064.
6. Bourissou, D.; Guerret, O.; Gabbai, F. P.; Bertrand, G.: Stable carbenes. Chem. Rev. **100** (2000) 39–91.
7. Patil, S.; Dietrich, K.; Deally, A.; Gleeson, B.; Müller-Bunz, H.; Paradisi, F.; Tacke, M.: Synthesis, cytotoxicity and antibacterial studies of novel symmetrically and nonsymmetrically 4-(methoxycarbonyl)benzyl-substituted *n*-heterocyclic carbene-silver acetate complexes. Helv. Chim. Acta **93** (2010) 2347–2364.
8. Hackenberg, F.; Lally, G.; Müller-Bunz, H.; Paradisi, F.; Quaglia, D.; Streciwilk, W.; Tacke, M.: Novel symmetrically *p*-benzyl-substituted 4,5-diaryl-imidazole *N*-heterocyclic carbene-silver(I) acetate complexes-synthesis and biological evaluation. J. Organomet. Chem. **717** (2012) 123–134.
9. Fang, Z.-L.; Zheng, S. R.; Fan, J.; Zhang, W.-G.: *In situ* synthesis of a zwitterionic ligand and formation of a Eu(III) coordination polymer with double-bridged connectors. Inorg. Chem. Commun. **20** (2012) 122–125.
10. Ennajih, H.; Bouhfid, R.; Zouihri, H.; Essassi, E. M.; Ng, S. W.: 1,3-Di-benzyl-2-methyl-benzimidazolium chloride. Acta Crystallogr. **E66** (2010) o455.