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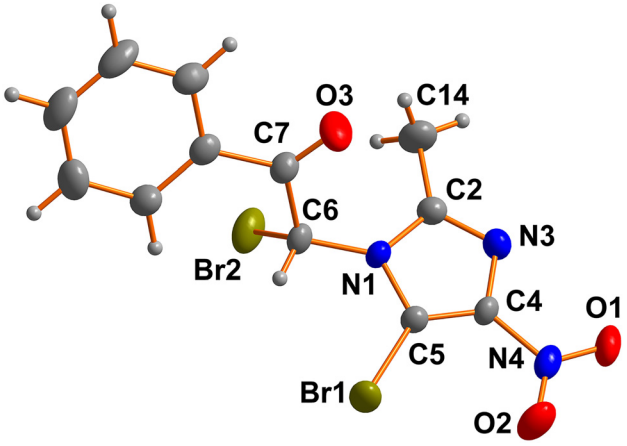
# The crystal structure of 2-bromo-2-(5-bromo-2-methyl-4-nitro-1*H*-imidazol-1-yl)-1-phenylethanone, $C_{12}H_9Br_2N_3O_3$

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**Abstract**

$C_{12}H_9Br_2N_3O_3$, monoclinic, $P2_1/c$ (no. 14), $a$ = 9.0255(5) Å, $b$ = 14.9112(5) Å, $c$ = 10.9438(5) Å, $\beta$ = 109.223(5)°, $V$ = 1390.71(12) Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0482, $wR_{ref}(F^2)$ = 0.0948, T = 293(2) K.

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The molecular 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.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Clear colourless block |
| Size: | 0.22 × 0.16 × 0.01 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 5.84 mm$^{-1}$ |
| Diffractometer, scan mode: | Xcalibur, Eos, $\varphi$ and $\omega$ scans |
| $\theta_{max}$, completeness: | 29.4°, 100 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 11361, 3325, 0.034 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2,459 |
| $N(param)_{refined}$: | 187 |
| Programs: | Bruker,[1] Olex2,[2] SHELX[3] |

## 1 Source of materials

The title compound was synthesized *via* double bromination of the precursor 2-(2-methyl-4-nitro-1*H*-imidazol-1-yl)-1-phenylethanone. The reaction was performed using liquid bromine in DMF, in the presence of potassium carbonate as a base. The new compound 2-bromo-2-(5-bromo-2-methyl-4-nitro-1*H*-imidazol-1-yl)-1-phenylethanone crystallized as off-white crystal in 84 % yield from dichloromethane-diethyl ether system. Melting Point: 176–178 °C. **HRMS–ESI** *m/z*: 401.89414 (M +H)$^+$. **$^1$H NMR** (500 MHz, DMSO-$d_6$, 298 K): $\delta$ 2.51 (*s*, 3H, H–C14); 7.51 (pseudo *t*, 2H, H–C10, H–C12); 7.63 (*t*, 1H, $J$ = 7.4 Hz, H–C11); 7.82 (*d*, 2H, $J$ = 7.7 Hz, H–C9, H–C13); 8.39 (*s*, 1H, H–C6). **$^{13}$C NMR** (125 MHz, DMSO-$d_6$, 298 K); $\delta$ 15.2 (C14); 59.7 (C6); 107.4 (C4); 129.2 (C9, C13); 129.4 (C10, C12); 132.9 (C11); 134.7 (C8); 144.9 (C2); 146.9 (C4); 187.3 (C7).

## 2 Experimental details

The chemicals were purchased from Aldrich (Germany), Fluka (Switzerland), and Scharlau, they were used without further purification, unless otherwise stated.

The NMR spectra were recorded on a Bruker Avance III-(500 MHz) spectrometer with tetramethylsilane (TMS) as an internal standard. $^1$H NMR (500 MHz), $^{13}$C NMR (125 MHz) were recorded in dimethylsulfoxide (DMSO-$d_6$). The following abbreviations are used to describe peak patterns: *s* = singlet, *t* = triplet, *d* = doublet. High-resolution mass spectra (HRMS) were measured (in positive/or negative ion

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.14800 (6) | 0.66340 (3) | 0.25408 (4) | 0.04043 (16) |
| Br2 | 0.05169 (6) | 0.37422 (4) | 0.10680 (5) | 0.04954 (18) |
| O3 | 0.4769 (4) | 0.4077 (2) | 0.3042 (3) | 0.0491 (9) |
| N1 | 0.2100 (4) | 0.4797 (2) | 0.3232 (3) | 0.0277 (8) |
| O1 | 0.1415 (4) | 0.5944 (2) | 0.6730 (3) | 0.0472 (9) |
| O2 | 0.1287 (5) | 0.6970 (2) | 0.5302 (3) | 0.0531 (10) |
| N3 | 0.2018 (4) | 0.4656 (2) | 0.5234 (3) | 0.0329 (8) |
| N4 | 0.1470 (4) | 0.6191 (3) | 0.5682 (3) | 0.0337 (8) |
| C4 | 0.1755 (5) | 0.5523 (3) | 0.4832 (4) | 0.0265 (9) |
| C6 | 0.2192 (5) | 0.4578 (3) | 0.1975 (4) | 0.0284 (9) |
| C5 | 0.1780 (5) | 0.5636 (2) | 0.3613 (4) | 0.0263 (9) |
| C8 | 0.4056 (5) | 0.4003 (3) | 0.0773 (4) | 0.0312 (9) |
| C2 | 0.2229 (5) | 0.4225 (3) | 0.4267 (4) | 0.0314 (9) |
| C7 | 0.3806 (5) | 0.4193 (3) | 0.2028 (4) | 0.0303 (9) |
| C9 | 0.5384 (6) | 0.3500 (3) | 0.0823 (5) | 0.0401 (11) |
| H9 | 0.6057 | 0.3294 | 0.1613 | 0.048* |
| C13 | 0.3082 (6) | 0.4323 (3) | −0.0414 (4) | 0.0367 (10) |
| H13 | 0.2207 | 0.4667 | −0.0455 | 0.044* |
| C12 | 0.3414 (7) | 0.4129 (3) | −0.1539 (5) | 0.0477 (13) |
| H12 | 0.2764 | 0.4342 | −0.2333 | 0.057* |
| C10 | 0.5691 (6) | 0.3313 (3) | −0.0301 (5) | 0.0490 (13) |
| H10 | 0.6569 | 0.2975 | −0.0269 | 0.059* |
| C11 | 0.4710 (7) | 0.3620 (3) | −0.1469 (5) | 0.0511 (14) |
| H11 | 0.4925 | 0.3484 | −0.2223 | 0.061* |
| C14 | 0.2489 (7) | 0.3238 (3) | 0.4275 (5) | 0.0509 (14) |
| H14A | 0.1708 | 0.2971 | 0.3547 | 0.076* |
| H14B | 0.2415 | 0.2986 | 0.5061 | 0.076* |
| H14C | 0.3513 | 0.3120 | 0.4224 | 0.076* |
| H6 | 0.201 (5) | 0.508 (3) | 0.152 (4) | 0.024 (11)* |

mode) using the electro spray ion trap (ESI po slow mass) technique by collision-induced dissociation on a Bruker APEX–IV (7 Tesla) instrument. Melting points were determined on a Stuart (SMP-10) melting point apparatus. Single crystal of $C_{12}H_9Br_2N_3O_3$ was obtained through crystallization of the pure compound from $CH_2Cl_2/Et_2O$. Using Olex2,[2] the structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL[4] refinement package. All H atoms bonded to C atoms were refined as riding, with CH distances of 0.93 Å (for aromatic ring).

## 3 Discussion

Imidazole forms the main structure of some well-known components of human organisms, that is, histidine, Vit–B12, a component of DNA base structure and purines, histamine and biotin. It is also present in the structure of many natural or synthetic drug molecules.[5] For example, lepidiline A and B exhibit significant cytotoxicity against various types of human cancer cell lines at micromolar concentrations.[6] Dacarbazine,[7] zoledronic acid[8] and azathioprine[9] are also potent anticancer agents bearing an imidazole ring. In consideration of the high therapeutic properties of the nitroimidazole-related drugs and in continuation of our research on the synthesis and biological evaluation of imidazole analogs,[10–16] we here present the important nucleus as imidazolyl containing title structure. Herein, the 2-bromo-1-phenylethane-1-one was coupled with imidazole ring to produce the target compound. The measurement shows that the title crystal structure consists of the monomeric molecules. In the title molecules all bond lengths are in normal ranges.[17] The average plane of imidazole ring is twisted (57.9(4)°) to the average plane of the ethanone group (C6 C7 C8). The shortest hydrogen intramolecular interactions are CH···O (C14H14A···O3, = 2.442(4) Å) and CH···Br interactions (C6H6···Br1 = 2.69(4) Å and C14H14C···Br2 = 2.8137(6) Å). The crystal is stabilized *via* CH…O (H10…O2 ($1 - x$, $-1/2 + y$, $1/2 - z$) = 2.457(4) Å) and CH···N (H12···N3 ($+x$, $+y$, $1 + z$) = 2.565(3) Å) interactions.

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