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Crystal structure of ethyl 1-(4-bromobenzyl)-3-phenyl-1H-pyrazole-5-carboxylate, C₁₉H₁₇BrN₂O₂

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Abstract
C₁₉H₁₇BrN₂O₂, triclinic, P ̅ ₁ (no. 2), a = 8.3979(11) Å, b = 10.4083(14) Å, c = 11.1696(15) Å, α = 69.270(2)°, β = 89.401(2)°, γ = 73.709(2)°, V = 872.1(2) Å³, Z = 2, Rgt(F) = 0.0422, wRref(F²) = 0.1138, T = 296.15 K.

Table 1: Data collection and handling.

<table>
<thead>
<tr>
<th>Crystal:</th>
<th>Block, colourless</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size:</td>
<td>0.20 × 0.20 × 0.15 mm</td>
</tr>
<tr>
<td>Wavelength:</td>
<td>Mo Kα radiation (0.71073 Å)</td>
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<tr>
<td>μ:</td>
<td>2.37 mm⁻¹</td>
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<tr>
<td>Diffractometer, scan mode:</td>
<td>Bruker P4, φ and ω-scans</td>
</tr>
<tr>
<td>θmax, completeness:</td>
<td>27.5°, &gt;92% (up to 25.2, &gt;99%)</td>
</tr>
<tr>
<td>N(hkl)measured, N(hkl)unique, Rint:</td>
<td>6989, 3699, 0.018</td>
</tr>
<tr>
<td>Criterion for Iobs, N(hkl)gt:</td>
<td>Iobs &gt; 2σ(Iobs), 2568</td>
</tr>
<tr>
<td>N(param)refined:</td>
<td>218</td>
</tr>
<tr>
<td>Programs:</td>
<td>Bruker programs [1], SHELX [2, 3]</td>
</tr>
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</table>

Discussion
Interest in pyrazole derivatives has increased during recent years due to their applications in various areas. Compounds with pyrazole core possess important biological and pharmacological properties such as antibacterial, anticancer, antihypertensive, cytotoxic, analgesic activities [4–8]. Hence, many pyrazole compounds with diversity and broad spectra of biological activity were reported [9–11]. In our previous work, we have synthesized a series of pyrazole derivatives with xanthine oxidase inhibitory activity [12, 13]. In order to increase the structural diversity of pyrazole with valuable biological properties, we report herein the crystal structure of ethyl 1-(4-bromobenzyl)-3-phenyl-1H-pyrazole-5-carboxylate.

In the title crystal structure, all bond lengths and angles are within normal ranges [14]. The bond lengths of N1—C8, N2—C6, C6—C7 and C7—C8 are 1.357(3), 1.341(4), 1.397(4) and 1.357(3) Å, respectively. The bond angles of C6—N1—C8, C6—N2—C8, and C6—N2—C7 are 116.2(2), 114.4(2), and 123.6(2)°, respectively. The bond lengths and angles of the title compound are within normal ranges, indicating that the compound has a stable structure in the crystal.

Source of material
To a solution of ethyl 3-phenyl-1H-pyrazole-5-carboxylate (0.52 g, 2.0 mmol) and K₂CO₃ (0.28 g, 2.0 mmol) in acetonitrile (10 mL) was added 4-bromobenzyl bromide (0.50 g, 2.0 mmol). The mixture was stirred at room temperature for 4 h and the precipitate was evaporated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using ethyl acetate and petroleum ether (1:5, v/v), to obtain the target compound in 94.4% yields.

Experimental details
All H atoms were placed in idealized positions and treated as riding on their parent atoms, with d(C—H) = 0.96 (methyl) and 0.97 Å (methylene), Uiso(H) = 1.5Ueq(C) and d(C—H) = 0.93 Å (aromatic), Uiso(H) = 1.2Ueq(C).
The dihedral angle between the aryl moiety (N1/C5/C4) and the aryl ring (C12/C13/C14/C15/C16/C17) show a dihedral angle of 71°. The dihedral angle between the aryl moiety (C1/C2/C3/C4/C18/C19) and pyrazole ring (N1/N2/C6/C7/C8) is 80.3°.

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References