Yunlu Li*

Crystal structure of 3-amino-N'-hydroxy-1H-pyrazole-4-carboximidamide, C₄H₇N₅O

Abstract

C₄H₇N₂O, monoclinic, P2₁ (no. 4), a = 4.855(3) Å, b = 9.026(5) Å, c = 7.092(4) Å, β = 103.267(7)°, V = 302.5(3) Å³, Z = 2, Rgt(F) = 0.0385, wRgt(F²) = 0.1183, T = 296(2) K. 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.

1 Source of materials

Using methanol as solvent, 2,4-dichloro-1,3,5-trinitrobenzene (0.70 g, 2.5 mmol) was mixed with 80 % hydrazine hydrate (0.75 ml, 18.8 mmol) at low temperature and continued to react at room temperature for 2 days. Then the reaction liquid was filtered, and the resulting filtrate was distilled under vacuum to obtain a yellow solid. The obtained solid was purified by silica gel column chromatography with ethyl acetate/petroleum ether mixture as eluent, and the yellow title compound crystals were obtained by slowly evaporating the solvent at room temperature.

Table 1: Data collection and handling.

<table>
<thead>
<tr>
<th>Crystal:</th>
<th>Colourless block</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size:</td>
<td>0.12 × 0.08 × 0.06 mm</td>
</tr>
<tr>
<td>Wavelength:</td>
<td>Mo Kα radiation (0.71073 Å)</td>
</tr>
<tr>
<td>μ:</td>
<td>0.12 mm⁻¹</td>
</tr>
<tr>
<td>Diffractometer, scan mode:</td>
<td>Bruker APEX-II, φ and ω</td>
</tr>
<tr>
<td>θmax, completeness:</td>
<td>27.6°, &gt;99 %</td>
</tr>
<tr>
<td>N(I( Państwowy)</td>
<td>1891, 1218, 0.021</td>
</tr>
<tr>
<td>Criterion for Iobs &gt; 2σ(Iobs), 1154</td>
<td></td>
</tr>
<tr>
<td>Programs:</td>
<td>97</td>
</tr>
</tbody>
</table>

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Ueq charismatic</th>
</tr>
</thead>
<tbody>
<tr>
<td>O1</td>
<td>0.8024 (4)</td>
<td>0.6764 (2)</td>
<td>0.9074 (3)</td>
<td>0.0352 (5)</td>
</tr>
<tr>
<td>H1</td>
<td>0.903 (8)</td>
<td>0.756 (4)</td>
<td>0.875 (2)</td>
<td>0.053*</td>
</tr>
<tr>
<td>N1</td>
<td>−0.1048 (5)</td>
<td>0.3263 (3)</td>
<td>0.4462 (3)</td>
<td>0.0301 (5)</td>
</tr>
<tr>
<td>N2</td>
<td>−0.0016 (5)</td>
<td>0.4043 (3)</td>
<td>0.3108 (3)</td>
<td>0.0279 (5)</td>
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<tr>
<td>N3</td>
<td>0.3688 (5)</td>
<td>0.5769 (3)</td>
<td>0.3277 (3)</td>
<td>0.0317 (5)</td>
</tr>
<tr>
<td>H3A</td>
<td>0.327301</td>
<td>0.588672</td>
<td>0.204078</td>
<td>0.038*</td>
</tr>
<tr>
<td>H3B</td>
<td>0.508847</td>
<td>0.625370</td>
<td>0.396600</td>
<td>0.038*</td>
</tr>
<tr>
<td>N4</td>
<td>0.5406 (5)</td>
<td>0.4349 (3)</td>
<td>0.9426 (3)</td>
<td>0.0360 (6)</td>
</tr>
<tr>
<td>H4A</td>
<td>0.667013</td>
<td>0.468942</td>
<td>1.038165</td>
<td>0.043*</td>
</tr>
<tr>
<td>H4B</td>
<td>0.456060</td>
<td>0.352855</td>
<td>0.954735</td>
<td>0.043*</td>
</tr>
<tr>
<td>N5</td>
<td>0.5891 (5)</td>
<td>0.6331 (3)</td>
<td>0.7419 (3)</td>
<td>0.0281 (5)</td>
</tr>
<tr>
<td>C1</td>
<td>0.2164 (5)</td>
<td>0.4804 (3)</td>
<td>0.4155 (3)</td>
<td>0.0229 (5)</td>
</tr>
<tr>
<td>C2</td>
<td>0.2553 (5)</td>
<td>0.4512 (3)</td>
<td>0.6156 (3)</td>
<td>0.0236 (5)</td>
</tr>
<tr>
<td>C3</td>
<td>0.0417 (5)</td>
<td>0.3519 (3)</td>
<td>0.6266 (4)</td>
<td>0.0282 (6)</td>
</tr>
<tr>
<td>H3</td>
<td>0.006903</td>
<td>0.310762</td>
<td>0.739080</td>
<td>0.034*</td>
</tr>
<tr>
<td>C4</td>
<td>0.4750 (5)</td>
<td>0.5097 (3)</td>
<td>0.7745 (3)</td>
<td>0.0240 (5)</td>
</tr>
<tr>
<td>H1A</td>
<td>−0.272 (8)</td>
<td>0.275 (4)</td>
<td>0.415 (5)</td>
<td>0.038 (9)*</td>
</tr>
</tbody>
</table>

2 Experimental details

The most hydrogen atoms were placed geometrically in calculated positions riding on attached atoms. C3-, N3- and N4-bound H atoms were refined as riding with Uiso(H) = 1.2Ueq. O1-bound H atom was refined as riding with Uiso(H) = 1.5Ueq. And the position of N1-bound H-atom was freely refined.

3 Comment

Amidoxime compounds are a kind of important chemical synthesis intermediates, which are widely used in the fields of...
organic luminescent materials, electron conversion materials, drug synthesis and the preparation of energetic compounds [5–8]. In the crystal structure of the title compound (cf. the figure), all bond lengths and angles are within the normal range [9–11]. And the molecular structure contains a pyrazole ring, an amino group and an amidoxime group, and their atoms respectively lie in separate planes. The dihedral angles of the pyrazole ring with the amino and amidoxime groups are 2.17° and 22.41°, respectively, so the molecule has a certain degree of planarity. And the structural characteristics of this compound are similar to those of 5-amino-N′-hydroxy-1H-1,2,3-triazole-4-carboximidamide [9] and 3-amino-1H-pyrazole-4-carbonitrile [10].

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**Conflict of interest statement:** The author declares no conflicts of interest regarding this article.

**References**