Crystal structures of $N$-[4-(trifluoromethyl)phenyl]benzamide and $N$-(4-methoxyphenyl)benzamide at 173 K: a study of the energetics of conformational changes due to crystal packing

Wayne H. Pearson,*‡ Joseph J. Urban, Amy H. Roy MacArthur, Shirley Lin and Dylan W. L. Cabrera

Chemistry Department, United States Naval Academy, 572 Holloway Rd, Annapolis, MD 21402, USA. *Correspondence e-mail: waynehp21662@gmail.com

As a part of our study of the syntheses of aryl amides, the crystal structures of two benzamides were determined from single-crystal X-ray data at 173 K. Both crystal structures contain molecular units as asymmetric units with no solvent in the unit cells. Crystal structure I, TFMP, is the result of the crystallization of $N$-[4-(trifluoromethyl)phenyl]benzamide, $C_{14}H_{10}F_{3}NO$. Crystal structure II, MOP, is composed of $N$-(4-methoxyphenyl)benzamide, $C_{14}H_{13}NO_{2}$, units. TFMP is triclinic, space group $P\overline{1}$, consisting of two molecules in the unit cell related by the center of symmetry. MOP is monoclinic, space group $P2_{1}/c$, consisting of four molecules in the unit cell. Both types of molecules contain three planar regions; a phenyl ring, an amide planar region, and a para-substituted phenyl ring. The orientations of these planar regions within the asymmetric units are compared to their predicted orientations, in isolation, from DFT calculations. The aryl rings are tilted approximately 60° with respect to each other in both experimentally determined structures, as compared to 30° in the DFT results. These conformational changes result in more favorable environments for N—H···O hydrogen bonding and aryl ring π-stacking in the crystal structures. Intermolecular interactions were examined by Hirshfeld surface analysis and quantified by calculating molecular interaction energies. The results of this study demonstrate that both hydrogen bonding and dispersion are essential to the side-by-side stacking of molecular units in these crystal structures. Weaker dispersion interactions along the axial directions of the molecules reveal insight into the melting mechanisms of these crystals.

1. Chemical context

Numerous methodologies have been developed to form amide C—N bonds due to their prevalence in biomolecules, such as peptides and proteins, and in synthetic targets (Seward & Jakubke, 2002; Greenberg et al., 2000). In particular, aryl amides can be found in a variety of pharmaceutical drugs and in polymers such as Kevlar® (Masse et al., 1998; Evano et al., 2004, 2008; Satyanarayana et al., 2007; Tanner et al., 1989). A series of aryl amides were synthesized and isolated during the development of a copper-mediated concurrent tandem catalytic methodology for the amidation of aryl chlorides (Chang et al., 2019). The crystal structures of two of these aryl amides, derived from the cross-coupling of either 4-chlorobenzotrifluoride or 4-chloroanisole with benzamide, are reported here.
2. Structural commentary

The reported compounds are substituted benzamides containing a para-substituted phenyl ring in place of one of the hydrogen atoms of the amide nitrogen. In both crystal structures, the asymmetric unit is a single molecule of the compound. Crystal structure I, TFMP, contains an asymmetric unit with a trifluoromethylphenyl ring. Crystal structure II, MOP, has an asymmetric unit with a methoxyphenyl ring. The molecular structures in the form of ellipsoid plots are shown in Fig. 1. There is nothing remarkable about the individual bond lengths, bond angles, or planarity of the aryl rings in these molecules.

Fig. 2 contains the unit cells for both crystal structures. Both molecules assume chiral configurations. Because the space groups are centrosymmetric, the unit-cell contents are racemic mixtures containing the enantiomers of the molecules in symmetry-related positions. In both crystal structures, the molecules align along the molecular axes. This alignment results in the long axes in both unit cells, \( c = 14.415(3) \) Å in TFMP and \( a = 26.783(2) \) Å in MOP.

![Figure 1](image1.png)

**Figure 1**
The molecules present in the asymmetric units in (a) TFMP and (b) MOP. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are represented by spheres of 0.20 Å radius.

![Figure 2](image2.png)

**Figure 2**
Unit-cell packing of (a) TFMP and (b) MOP.

Both molecules contain three planar regions; a phenyl ring, an amide linkage, and the para-substituted phenyl ring. Rotation of the rings relative to each other can lead to conformations that exist in the crystal structures that differ from the native molecular conformations. The relationship between the conformations of organic molecules and crystal structures has been reported extensively and summarized in the review article by Cruz-Cabeza & Bernstein (2014). Tilt angles were determined by comparing the angles between normals to least-squares planes as defined by the non-hydrogen atoms in a planar region. Significant tilt angles exist between the planar regions in both molecules in the experimentally determined structures as shown in Fig. 3.

3. DFT calculations and results for isolated molecules

Quantum-chemical density functional theory (DFT) calculations were performed to find the conformations of global minimum energy for the two molecules in isolation. Calculations were performed with the GAUSSIAN09 (Frisch et al., 2016) program suite on DoD High Performance Moderniza-
tion resources. Initial conformer searching was performed at the molecular mechanics level with the MMFF force field as implemented in SPARTAN molecular modeling software (Wavefunction, 2014). Viable structures were then subjected to complete geometry optimizations in GAUSSIAN09 at the M06-2X/6-31+G(d) level (Zhao & Truhlar, 2008). Frequency calculations were performed at M06-2X/6-31+G(d) to confirm that all stationary points were minima. Comparisons of bond lengths and angles between the experimentally determined structures and the DFT calculations can be found in the supporting information.

Tilts of the planar regions from the DFT calculations are also shown in Fig. 3. The amide plane/phenyl ring angles are approximately 29° in the experimental results and 27° in the calculated molecules. In the experimentally determined structures, the angles between para-substituted phenyl rings and the amide planes are 31.4 (2)° in TFMP and 38.4 (4)° in MOP. The DFT calculations yield much smaller angles of 8.5 and 7.9°, respectively. These results indicate that the conformational change due to crystal packing in both molecules is primarily due to ring tilts around the N1—C5 bonds while the rings joined by C8—C9 bonds are essentially oriented the same as in the isolated molecules. A search of benzamides in the Cambridge Structural Database (version 2020.3; Groom et al., 2016) revealed a number of compounds with similar phenyl ring/amide plane tilts. For example, N-phenylbenzamide (Wang et al., 2014), N-(4-hydroxyphenyl)benzamide (Tothadi & Desiraju, 2012), benzamide (Blake & Small, 1972) and N-(4-nitrophenyl)benzamide (du Plessis et al., 1983) all possess amide plane/phenyl ring angles between 28 and 31°. A likely explanation for the consistent amide plane/phenyl ring tilt would be the balance of the attractive O1⋯H14 interactions and the repulsive interactions of H1⋯H10.

Additional DFT calculations were performed to determine approximate energy differences between the molecules in isolation and conformations found in the crystal structures. To best approximate the conformations in the experimentally determined structures, dihedral angles around the amide linkage were constrained to crystallographic values while all other geometrical parameters were allowed to vary. Tilt angles between phenyl and para-substituted phenyl rings are in good agreement between the X-ray models and DFT calculations. For TFMP, the angles are 59.7 (1)° in the crystal structure and 59.6° in the DFT calculation. For MOP, the angles are 67.4 (1)° in the crystal structure and 66.8° in the DFT calculation. The results of the DFT calculations show that the energies of the conformations in the experimentally determined structures are slightly above those in the isolated molecules, viz. 3.2 kJ mol⁻¹ higher for TFMP and 2.5 kJ higher for MOP.

4. Supramolecular features

Close packing in both crystal structures is the result of hydrogen bonding, dipole interactions and dispersion. Hydrogen bonds were revealed by using the HTAB command in SHELXL (Sheldrick, 2015b) and verified using PLATON.

Figure 3

Views of orientations of planar regions and their dihedral angles (in °) from experimental results (top) and DFT calculations (bottom) for (a) TFMP and (b) MOP. Blue = phenyl ring; green = amide plane; mauve = para-substituted phenyl ring.
The H⋯O contacts are listed in Tables 1 and 2 and shown in Fig. 4. There is only one type of N—H⋯O interaction in both crystal structures, in the direction parallel to the a axis for TFMP and the b axis in MOP. There are non-classical carbon-based hydrogen bonds that exist as intra-molecular interactions (C6—H6⋯O1) in both crystal struc-

Table 1
Hydrogen-bond geometry (Å, °) for TFMP.

|          | D—H   | H⋯A   | D⋯A   | D—H⋯A |
|----------|--------|-------|-------|-------|
| C6—H6⋯O1 | 0.95   | 2.44  | 2.938 (4) | 112   |
| C4—H4⋯O1i| 0.95   | 2.57  | 3.240 (4) | 128   |
| N1—H1⋯O1i| 0.99 (1)| 2.23 (2) | 3.138 (3) | 151 (3) |

Symmetry code: (i) x, y, z.

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

|          | D—H   | H⋯A   | D⋯A   | D—H⋯A |
|----------|--------|-------|-------|-------|
| C6—H6⋯O1 | 0.95   | 2.49  | 2.912 (2) | 107   |
| N1—H1⋯O1i| 0.96 (1)| 2.16 (1) | 3.108 (2) | 166 (2) |

Symmetry code: (i) x, y−1, z.

Figure 4
Hydrogen bonding contacts (in Å) in (a) TFMP and (b) MOP.

Figure 5
Comparison of hydrogen-bonding environments (in Å) from X-ray results and DFT calculations for (a) TFMP and (b) MOP.
tures and intermolecular contacts (C4—H4 ⋅⋅⋅ O1) in TFMP only. The longer H4 ⋅⋅⋅ O1 contact in MOP (2.95 Å) is a result of the larger ring twist angle between the para-substituted phenyl ring and the amide linkage in MOP, 38.4° versus 31.4° in TFMP.

Comparisons of hydrogen-bonding regions from the experimentally determined structure and DFT results are shown in Fig. 5 for TFMP and MOP. In both cases, the molecules in the crystal structures have a more open environment with larger angles around the donor and acceptor sites and larger donor and acceptor cavities. The increased planar tilt between para-substituted phenyl rings and amide planes is a contributor to the more open hydrogen-bonding environments in the experimentally determined structures.

The increased tilt angles between the amide and para-substituted phenyl planes also facilitate the π-stacking in both crystal structures (Table 3). Neighboring environments around aryl rings are shown in Fig. 6. Each aryl ring has close contacts with six other aryl rings. In TFMP, there are contacts between trifluoromethylphenyl rings and phenyl rings. In MOP, phenyl rings have close contacts with phenyl rings while methoxy-phenyl rings have contacts with other methoxyphenyl rings on neighboring molecules. There are a total of six interactions surrounding each aryl ring, with four T-shaped interactions and two being a parallel displacement of rings. Neighboring molecules that have parallel displaced rings are involved in the N—H ⋅⋅⋅ O hydrogen bonding. A quantitative discussion of the π stacking geometries based upon the approach of Banerjee et al. (2019) can be found in the supporting information.

Table 3
π-stacking parameters for TFMP and MOP.
All distances are in Å with estimated uncertainties of 0.004. Angles are in ° with estimated uncertainties of 0.2.

| Centroid | Normal | Offset | Twist angle |
|----------|--------|--------|-------------|
| TFMP – surrounding both rings |
| 4.774 | 4.672 | 0.982 | 59.7 |
| 4.718 | 4.649 | 0.804 | 59.7 |
| 4.711 | 4.646 | 0.780 | 59.7 |
| 4.698 | 4.611 | 0.900 | 59.7 |
| 5.361 | 2.666 | 4.651 | 0.0 |
| MOP – surrounding phenyl rings |
| 4.781 | 4.757 | 0.478 | 64.6 |
| 4.901 | 4.875 | 0.504 | 64.6 |
| 5.248 | 2.802 | 4.437 | 0.0 |
| MOP – surrounding methoxyphenyl rings |
| 4.849 | 4.658 | 1.348 | 68.1 |
| 4.831 | 4.64 | 1.345 | 68.1 |
| 5.248 | 2.938 | 4.349 | 0.0 |

Figure 6
Hydrogen bonding and π-stacking (in Å) in (a) TFMP and (b) MOP (s.u.’s for centroid distances are approximately 0.005 Å). Riding H atoms are omitted for clarity.

Figure 7
View of contacts (in Å) along the molecular axes in (a) TFMP and (b) MOP.
5. Hirshfeld surfaces and molecular pair interaction energies

To further examine the supramolecular environments, Hirshfeld surfaces and molecular pair interaction energies were calculated for both crystal structures. All of these calculations were performed using the CE-B3LYP method via the TONTO program (Jayatilaka & Grimwood, 2003) as implemented in CrystalExplorer17 (Spackman et al., 2021). Interaction energies use benchmarked models based upon B3LYP/6-31G(d,p) functionals, coupled with appropriate scale factors for electrostatic, polarization, dispersion and repulsion energies. The CE-B3LYP model is benchmarked against B3LYP-D2/6-31G(d,p) counterpoise-corrected energies and has been found to give very good agreement with CCSD(T)/CBS (Turner et al., 2014).

Hirshfeld surfaces and molecular interaction energies are shown in Fig. 8. The neighboring molecules fall within 3.8 Å from the molecule inside the Hirshfeld surface. The color coding keys and scaled energies are found in Fig. 9. Although the energy values are reported to 0.1 of a kJ mol$^{-1}$, the authors of CrystalExplorer17 recommend that the reliability is on the order of 1 kJ mol$^{-1}$. As a result, the total interaction energies ($E_{\text{tot}}$) are rounded to a kJ mol$^{-1}$. As expected, the major $E_{\text{tot}}$ energies occur for the side-by-side interactions for TFMP (# 1–5) and MOP (# 1–3). The percent contributions to the $E_{\text{attract}}$ from the electrostatic, polarization and dispersion components are reported. Dispersion is the major attractive interaction in both crystal structures. For molecules with hydrogen-bonded close contacts and for some interactions along the molecular axes directions, the electrostatic energies are roughly equal to the dispersion energies. Videos showing 360° rotations of the static views in Fig. 8 can be found in the supporting information.

In Fig. 8, the electrostatic potentials, plotted on the Hirshfeld surfaces, show regions of negative charge (red) and positive charge (blue) for both compounds. For TFMP, in Fig. 8a, the electrostatic interaction of the hydrogen-bonding region is evident but so is the head-to-tail stacking of neighboring molecules due to the attraction of negative trifluoromethyl groups with neighboring positive phenyl hydrogens. For MOP, in Fig. 8b, the electrostatic interaction of the hydrogen bonding is apparent but the polar nature in the remaining segments of the molecule is localized in the methoxy group, contributing to the preference for association of methoxyphenyl rings in the crystal structure.

Fig. 8 also includes Hirshfeld surfaces with $d_{\text{norm}}$ surface plots. Intermolecular contacts less than a van der Waals contact are colored red, roughly equal contacts are white, and contacts longer than a van der Waals contact are blue. White or red contacts should indicate some degree of intermolecular interaction of inner and outer atoms at those positions on the Hirshfeld surfaces. Specific close contacts are shown in the supporting information.
Insight into the melting process for these crystals can be obtained from the energy analysis. Melting of these crystals would require overcoming the weak intermolecular interactions along the direction of the molecular axes. In the case of TFMP, the energy required would be on the order of 8–9 kJ mol\(^{-1}\) (interactions \#6 and \#7 in Fig. 9). In MOP, the energy required would only be around 7 kJ mol\(^{-1}\) (interactions \#5 and \#6). Although these energy values are internal energies and not enthalpies, they are reasonable values for heats of fusion and correlate with the melting points of the two crystal structures, 478 K for TFMP and 425 K for MOP (Chang et al., 2019). However, for TFMP, molecules should separate equally at the melting point on either side of a molecule. In MOP, molecules will separate first at the phenyl ends of the molecules while the methoxyphenyl ends would be predicted to persist into the liquid phase until enough energy was applied to overcome the 11 kJ mol\(^{-1}\) interaction energy (interaction \#4).

### 6. Database survey

The Cambridge Structural Database was searched for possible crystal structures of these compounds. No entries were found for a crystal structure of N-[4-(trifluoromethyl)phenyl]benzamide. A room-temperature crystal structure was found for the N-(4-methoxyphenyl)benzamide compound (du Plessis et al., 1983). The CIF file associated with this study, BUTDOJ, included only atom positions with no atomic displacement parameters. The \(R\) factor was listed as 0.106. In the published article, the authors noted that the overlapping reflections made it difficult to make an accurate background correction. This resulted, in the authors’ words, ‘in a somewhat poor set of intensity data for this compound’. For these reasons, we opted to use our redetermination of the crystal structure for the purpose of this publication.

### 7. Synthesis and crystallization

Details of the synthesis of the title compounds can be found in Chang et al. (2019). Product crystals for both compounds were grown by slow diffusion of hexanes into a concentrated solution of the amide in ethyl acetate.

### 8. Refinement

Crystal data, data collection and refinement details are summarized in Table 4. All hydrogen atoms were located in difference-Fourier maps. Final positions for most of the hydrogen atoms were calculated and included in a riding model relative to the bonded, non-hydrogen atoms by use of

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**Figure 9**

Key for the intermolecular interaction energies for TFMP and MOP. Energy units are kJ mol\(^{-1}\).

| Interaction | Color | \(R\) | \(E_{\text{ele}}\) | \(E_{\text{pol}}\) | \(E_{\text{disp}}\) | \(E_{\text{rep}}\) | \(E_{\text{tot}}\) | \(E_{\text{attract}}\) |
|-------------|-------|------|----------------|----------------|----------------|-------------|----------------|----------------|
| 1           |       | 5.36 | -31.7         | -5.1           | -34.3          | 24.5        | -47            | -71            |
| 2           |       | 5.26 | -13.6         | -1.6           | -39.4          | 14.5        | -40            | -55            |
| 3           |       | 5.17 | -12.4         | -2.2           | -42.2          | 18.5        | -38            | -57            |
| 4           |       | 6.32 | -13.4         | -1.3           | -37.0          | 14.7        | -37            | -52            |
| 5           |       | 6.28 | -10.9         | -2.1           | -37.5          | 18.0        | -33            | -50            |
| 6           |       | 10.68| -0.6          | -0.4           | -9.5           | 1.7         | -9             | -10            |
| 7           |       | 14.43| -3.3          | -0.4           | -7.8           | 3.0         | -8             | -11            |
| 8           |       | 14.42| -2.6          | -0.2           | -3.4           | 1.0         | -5             | -6             |
| 9           |       | 12.30| 2.3           | 0.2            | -4.6           | 0.4         | -2             | -5             |
| 10          |       | 11.95| 0.1           | 0.1            | -1.7           | 0.0         | -2             | -2             |
| 11          |       | 13.39| 0.1           | 0.1            | -1.2           | 0.0         | -1             | -1             |

| Interaction | Color | \(R\) | \(E_{\text{ele}}\) | \(E_{\text{pol}}\) | \(E_{\text{disp}}\) | \(E_{\text{rep}}\) | \(E_{\text{tot}}\) | \(E_{\text{attract}}\) |
|-------------|-------|------|----------------|----------------|----------------|-------------|----------------|----------------|
| 1           |       | 5.25 | -35.4         | -6.4           | -33.3          | 23.9        | -51            | -75            |
| 2           |       | 4.58 | -11.6         | -2.2           | -38.8          | 16.3        | -36            | -53            |
| 3           |       | 5.15 | -9.4          | -1.3           | -32.2          | 13.7        | -29            | -43            |
| 4           |       | 13.99| -8.0          | -0.9           | -7.3           | 4.8         | -11            | -16            |
| 5           |       | 13.45| -1.3          | -0.4           | -7.4           | 2.2         | -7             | -9             |
| 6           |       | 13.38| -2.1          | -0.1           | -7.8           | 3.5         | -7             | -10            |
| 7           |       | 14.20| 0.3           | -0.3           | -5.9           | 2.2         | -4             | -6             |
| 8           |       | 14.76| 0.2           | 0.1            | -3.0           | 0.9         | -2             | -3             |
| 9           |       | 14.17| -0.1          | 0.1            | -1.5           | 0.1         | -2             | -2             |

\(R = \text{distance between molecular centers}\)
\(E_{\text{ele}} = \text{electrostatic contribution}\)
\(E_{\text{pol}} = \text{polarization contribution}\)
\(E_{\text{disp}} = \text{dispersion contribution}\)
\(E_{\text{rep}} = \text{repulsion contribution}\)
\(E_{\text{tot}} = \text{total energy of interaction}\)
\(E_{\text{attract}} = \text{sum of negative contributions}\)
AFLIX commands. Methyl hydrogen atoms were fixed at 0.98 Å from bonded carbon atoms, and phenyl hydrogen atoms were located 0.95 Å from bonded carbon atoms. Hydrogen displacement parameters were isotropic and set at 1.20 times the bonded phenyl carbons and 1.50 times the bonded methyl carbon in MOP. The amide hydrogens were treated differently because of their participation in the hydrogen bonding in these crystal structures. DFIX commands were set at 1.00 Å for these hydrogen atoms to allow for better comparison with the DFT-calculated N—H bond lengths. These hydrogen positions were then refined with independent isotropic displacement parameters. Isotropic extinction was refined in MOP. Although the ‘standard’ independent atom model was used for our analysis, alternative models were considered. Refinements with librationaly corrected bond lengths and high-angle refinements were performed. These refinements had no significant effects on the structural results or the energy calculations.

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References
Banerjee, A., Saha, A. & Saha, B. K. (2019). Cryst. Growth Des. 19, 2245–2252.
Blake, C. C. F. & Small, R. W. H. (1972). Acta Cryst. B28, 2201–2206.
Bruker (2018). APEX3, SAINT and SADABS. Bruker AXS Inc, Madison, Wisconsin, USA.
Chang, R. K., Clairmont, B. P., Lin, S. & MacArthur, A. H. R. (2019). Organometallics. 38, 4448–4454.
Cruz-Cabeza, J. & Bernstein, J. (2014). Chem. Rev. 114, 2170–2191.
Evano, G., Blanchard, N. & Toumi, M. (2008). Chem. Rev. 108, 3054–3131.
Evano, G., Schaus, J. V. & Panek, J. S. (2004). Org. Lett. 6, 525–528.
Farrugia, L. J. (2012). J. Appl. Cryst. 45, 849–854.
Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Mennucci, B., Petersson, G. A., Nakatsuji, H., Caricato, M., Li, X., Hratchian, H. P., Izmaylov, A. F., Bloino, J., Zheng, G., Sonnenberg, J. L., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T.,

Table 4
Experimental details.

| Crystal data | TFMP | MOP |
|--------------|------|-----|
| Chemical formula | C$_{14}$H$_{10}$F$_{3}$NO | C$_{14}$H$_{13}$NO$_{2}$ |
| $M_r$ | 265.23 | 227.25 |
| Crystal system, space group | Triclinic, P$ar{T}$ | Monoclinic, P2$_1$/c |
| Temperature (K) | 173 | 173 |
| $a$, $b$, $c$ (Å) | 5.3606 (11), 7.7831 (16), 14.415 (3) | 26.7830 (15), 5.2477 (3), 8.1343 (5) |
| $\alpha$, $\beta$, $\gamma$ (°) | 77.170 (7), 79.421 (7), 89.719 (7) | 90, 97.594 (2), 90 |
| $V$ (Å$^3$) | 576.0 (2) | 1133.24 (11) |
| $Z$ | 2 | 4 |
| Radiation type | Mo Kα | Mo Kα |
| $\mu$ (mm$^{-1}$) | 0.13 | 0.09 |
| Crystal size (mm) | 0.24 × 0.08 × 0.06 | 0.70 × 0.26 × 0.08 |

| Data collection | TFMP | MOP |
|----------------|------|-----|
| Diffractometer | Bruker APEXII CCD | Bruker APEXII CCD |
| Absorption correction | Multi-scan (SADABS: Bruker, 2018) | Multi-scan (SADABS: Bruker, 2018) |
| $I_{	ext{min}}$, $I_{	ext{max}}$ | 0.700, 1.000 | 0.518, 1 |
| No. of measured, independent and observed | 11667, 2201, 1415 | 36318, 2333, 1678 |
| $R_{	ext{int}}$ | 0.086 | 0.106 |
| $\sin(\theta)_{\text{max}}$ (Å$^{-1}$) | 0.612 | 0.626 |

| Refinement | TFMP | MOP |
|-------------|------|-----|
| $R(F^2 > 2\sigma(F^2))$, $wR(F^2)$, $S$ | 0.068, 0.189, 1.11 | 0.049, 0.126, 1.08 |
| No. of reflections | 2201 | 2333 |
| No. of parameters | 176 | 160 |
| No. of restraints | 1 | 1 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Deltaho_{	ext{max}}$, $\Deltaho_{	ext{min}}$ (e Å$^{-3}$) | 0.26, −0.32 | 0.20, −0.19 |

Computer programs: APEX3 and SAINT (Bruker, 2018), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), WinGX (Farrugia, 2012), Mercury (Macrae et al., 2020), CrystalExplorer17 (Spackman et al., 2021), and publCIF (Westrip, 2010).
Montgomery, J. A., Jr., Peralta, J. E., Ogliaro, F., Bearpark, M., Heyd, J. J., Brothers, E., Kudin, K. N., Staroverov, V. N., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Rega, N., Millam, J. M., Klene, M., Knox, J. E., Cross, J. B., Bakken, V., Adamo, C., Jaramillo, J., Gomperts, R., Stratmann, R. E., Yazyev, O., Austin, A. J., Cammi, R., Pomelli, C., Ochterski, J. W., Martin, R. L., Morokuma, K., Zakrzewski, V. G., Voth, G. A., Salvador, P., Dannenberg, J. J., Dapprich, S., Daniels, A. D., Farkas, O., Foresman, J. B., Ortiz, J. V., Cioslowski, J. & Fox, D. J. (2016). *Gaussian 09*, Revision A. 02. Gaussian Inc., Wallingford CT, USA.

Greenberg, A., Breneman, C. M. & Liebman, J. F. (2000). *Amide Linkage: Selected Structural Aspects in Chemistry, Biochemistry, and Materials Science*. New York: Wiley-Interscience.

Groom, C. R., Bruno, I. J., Lightfoot, M. F. & Ward, S. C. (2016). *Acta Cryst.* B72, 171–179.

Jayatilaka, D. & Grimwood, D. J. (2003). *Computational Science|ICCS 2003*, edited by P. M. A. Sloot, D. Abramson, A. V. Bogdanov, Y. E. Gorbachev, J. J. Dongarra & A. Y. Zomaya, pp. 142–151. Berlin, Heidelberg: Springer.

Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* 53, 226–235.

Masse, C. E., Yang, M., Soloman, J. & Panek, J. S. (1998). *J. Am. Chem. Soc.* 120, 4123–4134.

Plessis, M. P. du, Modro, T. A. & Nassimbeni, L. R. (1983). *J. Crystallogr. Spectrosc. Res.* 13, 179–189.

Satyanarayana, K., Srinivas, K., Himabindu, V. & Reddy, G. M. (2007). *Org. Process Res. Dev.* 11, 842–845.

Seward, N. & Jakubke, H. D. (2002). *Peptides: Chemistry and Biology*. Weinheim: Wiley-VCH.

Sheldrick, G. M. (2015a). *Acta Cryst.* A71, 3–8.

Sheldrick, G. M. (2015b). *Acta Cryst.* C71, 3–8.

Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). *J. Appl. Cryst.* 54, 1006–1011.

Spek, A. L. (2020). *Acta Cryst.* E76, 1–11.

Tanner, D., Fitzgerald, J. A. & Phillips, B. R. (1989). *Angew. Chem. Int. Ed. Engl.* 28, 649–654.

Tothadi, S. & Desiraju, G. R. (2012). *Cryst. Growth Des.* 12, 6188–6198.

Turner, M. J., Grabowsky, S., Jayatilaka, D. & Spackman, M. A. (2014). *J. Phys. Chem. Lett.* 5, 4229–4255.

Wang, J.-L., Xu, J.-S., Wang, D.-Y., Wang, H., Li, Z.-T. & Zhang, D.-W. (2014). *CrystEngComm*, 16, 2078–2084.

Wavefunction (2014). *SPARTAN*. Wavefunction Inc., Irvine, CA, USA.

Westrip, S. P. (2010). *J. Appl. Cryst.* 43, 920–925.

Zhao, Y. & Truhlar, D. J. (2008). *Theor. Chem. Acc.* 120, 215–241.
Crystal structures of \(N\)-[4-(trifluoromethyl)phenyl]benzamide and \(N\)-(4-methoxyphenyl)benzamide at 173 K: a study of the energetics of conformational changes due to crystal packing

Wayne H. Pearson, Joseph J. Urban, Amy H. Roy MacArthur, Shirley Lin and Dylan W. L. Cabrera

Computing details

For both structures, data collection: \textit{APEX3} (Bruker, 2018); cell refinement: \textit{SAINT} (Bruker, 2018); data reduction: \textit{SAINT} (Bruker, 2018); program(s) used to solve structure: \textit{SHELXT} (Sheldrick, 2015a); program(s) used to refine structure: \textit{WinGX} (Farrugia, 2012), \textit{SHELXL} (Sheldrick, 2015b); molecular graphics: \textit{Mercury} (Macrae et al., 2020) and \textit{CrystalExplorer17} Spackman et al., 2021); software used to prepare material for publication: \textit{publCIF} (Westrip, 2010).

\(N\)-[4-(Trifluoromethyl)phenyl]benzamide (1)

\textit{Crystal data}

\(\text{C}_{14}\text{H}_{10}\text{F}_{3}\text{NO}\)
\(M_r = 265.23\)
Triclinic, \(\text{P}\bar{1}\)
\(a = 5.3606 (11) \text{ Å}\)
\(b = 7.7831 (16) \text{ Å}\)
\(c = 14.415 (3) \text{ Å}\)
\(\alpha = 77.170 (7)^{\circ}\)
\(\beta = 79.421 (7)^{\circ}\)
\(\gamma = 89.719 (7)^{\circ}\)
\(V = 576.0 (2) \text{ Å}^3\)
\(Z = 2\)
\(F(000) = 272\)

\textit{Data collection}

Bruker APEXII CCD
diffractometer
Radiation source: sealed X-ray tube
Detector resolution: 8.53 pixels mm\(^{-1}\)
rotating crystal scans
Absorption correction: multi-scan
\(\text{(SADABS; Bruker, 2018)}\)
\(T_{\text{min}} = 0.700, T_{\text{max}} = 1.000\)

\(D_x = 1.529 \text{ Mg m}^{-3}\)
\(D_m = 1.46 \text{ (2) Mg m}^{-3}\)
\(D_m\) measured by flotation in K2CO3(aq)
solution
Melting point = 477–478 K
Mo \(K\alpha\) radiation, \(\lambda = 0.71073 \text{ Å}\)
Cell parameters from 2253 reflections
\(\theta = 2.7–23.2^\circ\)
\(\mu = 0.13 \text{ mm}^{-1}\)
\(T = 173 \text{ K}\)
Regular parallelepiped, colourless
\(0.24 \times 0.08 \times 0.06 \text{ mm}\)

11667 measured reflections
2201 independent reflections
1415 reflections with \(I > 2\sigma(I)\)
\(R_{\text{int}} = 0.086\)
\(\theta_{\text{max}} = 25.8^\circ, \theta_{\text{min}} = 1.5^\circ\)
\(h = -6\rightarrow 6\)
\(k = -9\rightarrow 9\)
\(l = -17\rightarrow 17\)
Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.068$

$wR(F^2) = 0.189$

$S = 1.11$

2201 reflections

176 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: structure-invariant direct methods

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0726P)^2 + 0.6085P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.26 e\,\text{Å}^{-3}$

$\Delta\rho_{\text{min}} = -0.32 e\,\text{Å}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)

|   | $x$    | $y$    | $z$    | $U_{iso}^{*}\text{ Å}^2$ | $U_{eq}\text{ Å}^2$ |
|---|--------|--------|--------|--------------------------|---------------------|
| C1| 0.4290 (8) | 0.2664 (5) | 0.9270 (3) | 0.0361 (10) |
| C2| 0.5101 (6) | 0.2716 (5) | 0.8223 (3) | 0.0259 (8) |
| C3| 0.3572 (6) | 0.3479 (5) | 0.7572 (3) | 0.0274 (9) |
| H3| 0.207249 | 0.403788 | 0.779424 | 0.033* |
| C4| 0.4208 (6) | 0.3432 (5) | 0.6611 (3) | 0.0269 (8) |
| C5| 0.314467 | 0.395672 | 0.617458 | 0.032* |
| C6| 0.6407 (6) | 0.2620 (4) | 0.6270 (2) | 0.0228 (8) |
| C7| 0.7958 (6) | 0.1861 (5) | 0.6926 (3) | 0.0260 (8) |
| C8| 0.947277 | 0.131744 | 0.670270 | 0.031* |
| C9| 0.7301 (6) | 0.1899 (5) | 0.7885 (2) | 0.0253 (8) |
| H9| 0.834978 | 0.136384 | 0.832438 | 0.030* |
| C10| 0.9209 (6) | 0.2491 (5) | 0.4709 (3) | 0.0257 (8) |
| C11| 0.9138 (6) | 0.2390 (5) | 0.3692 (3) | 0.0238 (8) |
| C12| 0.7131 (6) | 0.1552 (5) | 0.3439 (3) | 0.0265 (8) |
| C13| 0.572074 | 0.103421 | 0.392070 | 0.032* |
| C14| 0.7225 (6) | 0.1487 (5) | 0.2481 (3) | 0.0302 (9) |
| H11| 0.587155 | 0.091657 | 0.230932 | 0.036* |
| C12| 0.9254 (7) | 0.2236 (5) | 0.1773 (3) | 0.0304 (9) |
| C13| 0.928932 | 0.218903 | 0.111750 | 0.036* |
| C14| 1.1249 (7) | 0.3062 (5) | 0.2021 (3) | 0.0303 (9) |
| C15| 1.265418 | 0.357914 | 0.153574 | 0.036* |
| C16| 1.1180 (6) | 0.3127 (5) | 0.2979 (3) | 0.0280 (9) |
| C17| 1.255099 | 0.368467 | 0.314711 | 0.034* |
| F1| 0.3071 (5) | 0.4105 (3) | 0.94336 (17) | 0.0520 (7) |
| F2| 0.6234 (5) | 0.2548 (4) | 0.97444 (18) | 0.0599 (8) |
| F3| 0.2713 (5) | 0.1294 (4) | 0.97311 (18) | 0.0606 (8) |
| N1| 0.6886 (5) | 0.2533 (4) | 0.5288 (2) | 0.0253 (7) |
| O1| 1.1224 (4) | 0.2563 (4) | 0.49980 (18) | 0.0359 (7) |
| H1| 0.541 (5) | 0.261 (6) | 0.495 (3) | 0.052 (13)* |
### Atomic displacement parameters (Å²)

|       | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-------|-----------|-----------|-----------|-----------|-----------|-----------|
| C1    | 0.039 (2) | 0.033 (2) | 0.037 (2) | 0.0094 (19) | −0.0088 (18) | −0.0111 (18) |
| C2    | 0.0212 (17) | 0.027 (2) | 0.030 (2) | 0.0040 (15) | −0.0059 (15) | −0.0067 (16) |
| C3    | 0.0187 (17) | 0.031 (2) | 0.033 (2) | 0.0076 (15) | −0.0031 (15) | −0.0104 (17) |
| C4    | 0.0178 (17) | 0.030 (2) | 0.036 (2) | 0.0046 (15) | −0.0092 (15) | −0.0095 (16) |
| C5    | 0.0179 (16) | 0.022 (2) | 0.029 (2) | 0.0015 (14) | −0.0057 (14) | −0.0065 (15) |
| C6    | 0.0159 (17) | 0.032 (2) | 0.031 (2) | 0.0082 (15) | −0.0052 (14) | −0.0077 (16) |
| C7    | 0.0204 (17) | 0.027 (2) | 0.029 (2) | 0.0061 (15) | −0.0095 (15) | −0.0031 (15) |
| C8    | 0.0196 (18) | 0.026 (2) | 0.032 (2) | 0.0036 (14) | −0.0044 (15) | −0.0080 (16) |
| C9    | 0.0191 (17) | 0.024 (2) | 0.0293 (19) | 0.0114 (14) | −0.0054 (14) | −0.0070 (15) |
| C10   | 0.0158 (16) | 0.029 (2) | 0.035 (2) | 0.0062 (14) | −0.0043 (14) | −0.0084 (16) |
| C11   | 0.0206 (18) | 0.034 (2) | 0.040 (2) | 0.0090 (16) | −0.0123 (16) | −0.0127 (17) |
| C12   | 0.029 (2) | 0.037 (2) | 0.030 (2) | 0.0127 (17) | −0.0098 (16) | −0.0119 (17) |
| C13   | 0.0233 (19) | 0.033 (2) | 0.032 (2) | 0.0101 (16) | −0.0007 (15) | −0.0054 (17) |
| C14   | 0.0180 (17) | 0.032 (2) | 0.036 (2) | 0.0071 (15) | −0.0073 (15) | −0.0097 (17) |
| F1    | 0.0738 (18) | 0.0462 (16) | 0.0401 (14) | 0.0300 (13) | −0.0083 (12) | −0.0207 (12) |
| F2    | 0.0573 (16) | 0.093 (2) | 0.0404 (15) | 0.0265 (15) | −0.0240 (13) | −0.0268 (14) |
| F3    | 0.0735 (19) | 0.0528 (17) | 0.0442 (16) | −0.0077 (14) | 0.0159 (13) | −0.0089 (12) |
| N1    | 0.0147 (14) | 0.0351 (19) | 0.0279 (17) | 0.0076 (13) | −0.0064 (12) | −0.0093 (13) |
| O1    | 0.0162 (12) | 0.062 (2) | 0.0331 (15) | 0.0062 (12) | −0.0074 (11) | −0.0161 (13) |

### Geometric parameters (Å, °)

|         | C1—F3          | C8—O1          | 1.233 (4) |
|---------|----------------|----------------|-----------|
| C1—F2  | 1.340 (4)       | C8—N1          | 1.370 (4) |
| C1—F1  | 1.340 (4)       | C8—C9          | 1.493 (5) |
| C1—C2  | 1.484 (5)       | C9—C14         | 1.384 (5) |
| C2—C3  | 1.391 (5)       | C9—C10         | 1.405 (5) |
| C2—C7  | 1.397 (5)       | C10—C11        | 1.385 (5) |
| C3—C4  | 1.373 (5)       | C10—H10        | 0.9500    |
| C3—H3  | 0.9500          | C11—C12        | 1.378 (5) |
| C4—C5  | 1.396 (5)       | C11—H11        | 0.9500    |
| C4—H4  | 0.9500          | C12—C13        | 1.391 (5) |
| C5—C6  | 1.403 (5)       | C12—H12        | 0.9500    |
| C5—N1  | 1.409 (4)       | C13—C14        | 1.387 (5) |
| C6—C7  | 1.370 (5)       | C13—H13        | 0.9500    |
| C6—H6  | 0.9500          | C14—H14        | 0.9500    |
| C7—H7  | 0.9500          | N1—H1          | 0.993 (10) |

F3—C1—F2 105.9 (3) O1—C8—N1 122.7 (3)
C3—C2—C1 120.0 (3)  C11—C10—H10 120.3
C7—C2—C1 120.9 (3)  C9—C10—H10 120.3
C4—C3—C2 120.7 (3)  C12—C11—C10 121.0 (3)
C4—C3—H3 119.7  C12—C11—H11 119.5
C2—C3—H3 119.7  C10—C11—H11 119.5
C3—C4—C5 120.5 (3)  C11—C12—C13 119.8 (3)
C4—C3—H3 119.7  C12—C11—H11 121.0
C5—C4—H4 119.8  C13—C12—H12 120.1
C4—C5—C6 118.9 (3)  C14—C13—C12 119.7 (3)
C4—C5—N1 117.8 (3)  C14—C13—H13 120.1
C6—C5—N1 123.3 (3)  C12—C13—H13 120.1
C7—C6—C5 120.4 (3)  C9—C14—C13 120.8 (3)
C7—C6—H6 119.8  C9—C14—H14 119.6
C5—C6—H6 119.8  C14—C13—H14 119.6
C6—C7—C2 120.6 (3)  C8—N1—C5 127.0 (3)
C6—C7—H7 119.7  C8—N1—H1 120.1
C2—C7—H7 119.7  C5—N1—H1 120.1

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| C6—H6···O1 | 0.95 | 2.44 | 2.938 (4) | 112 |
| C4—H4···O1i | 0.95 | 2.57 | 3.240 (4) | 128 |
| N1—H1···O1i | 0.99 (1) | 2.23 (2) | 3.138 (3) | 151 (3) |

Symmetry code: (i) x, y, z.

N-(4-Methoxyphenyl)benzamide (2)

Crystal data

C_{14}H_{13}NO_{2}  
M_r = 227.25
Monoclinic, P2_{1}/c
a = 26.7830 (15) Å  
b = 5.2477 (3) Å  
c = 8.1343 (5) Å  
β = 97.594 (2)°  
V = 1133.24 (11) Å³  
Z = 4
F(000) = 480

Data collection

Bruker APEXII CCD diffractometer  
Radiation source: sealed X-ray tube  
Detector resolution: 8.53 pixels mm⁻¹  
rotating crystal scans  
Absorption correction: multi-scan (SADABS; Bruker, 2018)  
T_{min} = 0.518, T_{max} = 1

36318 measured reflections  
2333 independent reflections

θ_{max} = 26.4°, θ_{min} = 1.5°  
h = −33→33  
k = −6→6  
l = −10→10

Mo Kα radiation, λ = 0.71073 Å  
Cell parameters from 5514 reflections  
θ = 3.1−26.0°  
µ = 0.09 mm⁻¹  
T = 173 K  
Regular parallelepiped, colourless  
0.70 × 0.26 × 0.08 mm

Melting point = 424–425 K  
D_{m} measured by flotation in aqueous KI  
D_{m} = 1.29 (2) Mg m⁻³  
D_{m} = 1.332 Mg m⁻³  

Acta Cryst. (2022). E78, 297-305
**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R(F^2) = 0.049$

$wR(F^2) = 0.126$

$S = 1.08$

2333 reflections

160 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: structure-invariant direct methods

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F^2) + (0.0247P)^2 + 0.8083P]$

where $P = (F_c^2 + 2F_s^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.20 \text{ e Å}^{-3}$

$\Delta\rho_{\text{min}} = -0.19 \text{ e Å}^{-3}$

Extinction correction: SHELXL-2018/3 (Sheldrick, 2015b), $F^c=kF^c[1+0.001xF^2/\lambda^2/sin(2\theta)]^{1/4}$

Extinction coefficient: 0.0099 (18)

Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** isotropic extinction correction applied

| Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\AA^2$) |
|---|---|---|---|---|
| C1 | 0.53199 (8) | 0.7401 (5) | 0.1647 (3) | 0.0544 (7) |
| H1A | 0.529249 | 0.745250 | 0.283557 | 0.082* |
| H1B | 0.498295 | 0.724788 | 0.101786 | 0.082* |
| H1C | 0.547952 | 0.897114 | 0.132379 | 0.082* |
| C2 | 0.61069 (7) | 0.5196 (4) | 0.2084 (2) | 0.0350 (5) |
| C3 | 0.63993 (8) | 0.3201 (4) | 0.1635 (3) | 0.0388 (5) |
| H3 | 0.625795 | 0.197660 | 0.084749 | 0.047* |
| C4 | 0.68972 (8) | 0.2992 (4) | 0.2334 (3) | 0.0369 (5) |
| H4 | 0.709605 | 0.161154 | 0.203434 | 0.044* |
| C5 | 0.71075 (7) | 0.4793 (4) | 0.3471 (2) | 0.0319 (4) |
| C6 | 0.68127 (8) | 0.6755 (4) | 0.3937 (3) | 0.0362 (5) |
| H6 | 0.695284 | 0.796302 | 0.473747 | 0.043* |
| C7 | 0.63122 (7) | 0.6971 (4) | 0.3240 (3) | 0.0361 (5) |
| H7 | 0.611137 | 0.833235 | 0.355679 | 0.043* |
| C8 | 0.79514 (7) | 0.6427 (4) | 0.4517 (3) | 0.0367 (5) |
| C9 | 0.84768 (7) | 0.5666 (4) | 0.5223 (3) | 0.0343 (5) |
| C10 | 0.85869 (8) | 0.3512 (4) | 0.6189 (3) | 0.0412 (5) |
| H10 | 0.832343 | 0.240278 | 0.640884 | 0.049* |
| C11 | 0.90810 (9) | 0.2974 (5) | 0.6833 (3) | 0.0493 (6) |
| H11 | 0.915465 | 0.151421 | 0.751318 | 0.059* |
| C12 | 0.94644 (9) | 0.4548 (5) | 0.6491 (3) | 0.0502 (6) |
| H12 | 0.980294 | 0.416164 | 0.692929 | 0.060* |
| C13 | 0.93588 (8) | 0.6682 (5) | 0.5525 (3) | 0.0495 (6) |
| H13 | 0.962451 | 0.776471 | 0.528736 | 0.059* |
| C14 | 0.88664 (8) | 0.7252 (4) | 0.4899 (3) | 0.0417 (5) |
| H14 | 0.879440 | 0.873678 | 0.424217 | 0.050* |
N1 0.76211 (6) 0.4491 (3) 0.4173 (2) 0.0358 (4)
O2 0.56174 (5) 0.5266 (3) 0.13011 (19) 0.0477 (4)
O1 0.78412 (6) 0.8674 (3) 0.4262 (2) 0.0572 (5)
H1 0.7748 (8) 0.277 (2) 0.424 (3) 0.041 (6)*

Atomic displacement parameters ( Å²)

|     | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-----|----------|----------|----------|----------|----------|----------|
| C1  | 0.0349 (12) | 0.0616 (16) | 0.0642 (17) | 0.0071 (11) | −0.0026 (11) | −0.0003 (13) |
| C2  | 0.0296 (10) | 0.0395 (11) | 0.0348 (11) | −0.0032 (9) | 0.0001 (8) | 0.0042 (9) |
| C3  | 0.0404 (12) | 0.0366 (12) | 0.0377 (12) | −0.0039 (9) | −0.0014 (9) | −0.0053 (9) |
| C4  | 0.0365 (11) | 0.0321 (11) | 0.0420 (12) | 0.0014 (9) | 0.0053 (9) | −0.0020 (9) |
| C5  | 0.0310 (10) | 0.0309 (10) | 0.0334 (11) | −0.0017 (8) | 0.0024 (8) | 0.0033 (8) |
| C6  | 0.0347 (11) | 0.0341 (11) | 0.0382 (12) | −0.0002 (9) | −0.0010 (9) | −0.0050 (9) |
| C7  | 0.0323 (11) | 0.0356 (11) | 0.0402 (12) | 0.0021 (9) | 0.0044 (9) | −0.0026 (9) |
| C8  | 0.0331 (11) | 0.0313 (11) | 0.0447 (13) | 0.0016 (9) | 0.0020 (9) | −0.0001 (9) |
| C9  | 0.0321 (11) | 0.0316 (10) | 0.0380 (11) | 0.0013 (8) | 0.0007 (9) | −0.0042 (9) |
| C10 | 0.0410 (12) | 0.0359 (12) | 0.0449 (13) | −0.0010 (9) | −0.0010 (10) | 0.0002 (10) |
| C11 | 0.0505 (14) | 0.0432 (13) | 0.0504 (14) | 0.0103 (11) | −0.0079 (11) | 0.0015 (11) |
| C12 | 0.0349 (12) | 0.0584 (15) | 0.0539 (15) | 0.0113 (11) | −0.0066 (10) | −0.0111 (12) |
| C13 | 0.0336 (12) | 0.0557 (15) | 0.0576 (15) | −0.0045 (11) | 0.0009 (10) | −0.0062 (12) |
| C14 | 0.0365 (11) | 0.0394 (12) | 0.0479 (13) | −0.0023 (9) | 0.0011 (10) | 0.0016 (10) |
| N1  | 0.0297 (9) | 0.0281 (9) | 0.0479 (11) | 0.0017 (7) | −0.0014 (7) | 0.0007 (8) |
| O2  | 0.0301 (8) | 0.0552 (10) | 0.0548 (10) | 0.0004 (7) | −0.0060 (7) | −0.0063 (8) |
| O1  | 0.0376 (9) | 0.0302 (8) | 0.0996 (14) | 0.0016 (7) | −0.0063 (9) | 0.0055 (9) |

Geometric parameters (Å, °)

|     | C1—O2     | C8—O1     | C1—H1A   | C8—N1     | C1—H1B   | C8—C9     | C1—H1C   | C9—C10   | C2—O2     | C9—C14   | C2—C7     | C10—C11   | C2—C3     | C10—H10   | C3—C4     | C11—C12   | C3—H3     | C11—H11   | C4—C5     | C12—C13   | C4—H4     | C12—H12   | C5—C6     | C13—C14   | C5—N1     | C13—H13   | C6—C7     | C14—H14   | C6—H6     | N1—H1     | C7—H7     | O2—C1     | H1A—C1     |
|-----|------------|------------|----------|------------|----------|------------|----------|----------|------------|----------|------------|------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
|     | 1.424 (3)  | 1.227 (2)  | 0.9800   | 1.352 (3)  | 0.9800   | 1.502 (3)  | 0.9800   | 1.386 (3) | 1.380 (2)  | 1.387 (3) | 1.385 (3)  | 1.387 (3)  | 1.385 (3) | 0.9500   | 1.389 (3) | 1.376 (3) | 0.9500   | 1.381 (3) | 1.382 (3) | 1.426 (2) | 0.9500   | 1.389 (3) | 0.9500   | 1.426 (2) | 0.9500   | 1.397 (3) | 0.9500   |
|     | O2—C1—H1A | 109.5      | O1—C8—C9| 120.83 (18)| O2—C1—H1B| 109.5      | N1—C8—C9| 111.52 (17)| O1—C1—H1B| 109.5      | C10—C9—C14| 119.21 (19)|
O2—C1—H1C 109.5  C10—C9—C8 123.50 (19)
H1A—C1—H1C 109.5  C14—C9—C8 117.28 (19)
H1B—C1—H1C 109.5  C9—C10—C11 120.1 (2)
O2—C2—C7 124.29 (19)  C9—C10—H10 119.9
O2—C2—C3 115.73 (18)  C11—C10—H10 119.9
C7—C2—C3 119.98 (18)  C12—C11—C10 120.1 (2)
C4—C3—C2 119.97 (19)  C12—C11—H11 119.9
C4—C3—H3 120.0  C10—C11—H11 119.9
C2—C3—H3 120.0  C11—C12—C13 120.1 (2)
C3—C4—C5 120.28 (19)  C11—C12—H12 119.9
C3—C4—H4 119.9  C13—C12—H12 119.9
C5—C4—H4 119.9  C12—C13—C14 120.0 (2)
C6—C5—C4 119.58 (18)  C12—C13—H13 120.0
C6—C5—N1 121.95 (18)  C14—C13—H13 120.0
C4—C5—N1 118.42 (18)  C13—C14—C9 120.4 (2)
C5—C6—C7 120.34 (19)  C13—C14—H14 119.8
C5—C6—H6 119.8  C9—C14—H14 119.8
C7—C6—H6 119.8  C8—N1—C5 124.71 (17)
C2—C7—C6 119.81 (19)  C8—N1—H1 118.4 (13)
C2—C7—H7 120.1  C5—N1—H1 116.2 (13)
C6—C7—H7 120.1  C2—O2—C1 116.87 (17)
O1—C8—N1 123.44 (19)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|----------|------|-------|-------|---------|
| C6—H6···O1 | 0.95 | 2.49 | 2.912 (2) | 107 |
| N1—H1···O1 | 0.96 (1) | 2.16 (1) | 3.108 (2) | 166 (2) |

Symmetry code: (i) x, y−1, z.