Syntheses and crystal structures of 4-(4-nitrophenyl)piperazin-1-ium benzoate monohydrate and 4-(4-nitrophenyl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate

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Crystal structures are reported for two molecular salts containing the 4-(4-nitrophenyl)piperazin-1-ium cation. Co-crystallization from methanol/ethyl acetate solution of N-(4-nitrophenyl)piperazine with benzoic acid gives the benzoate salt, which crystallizes as a monohydrate, C_{10}H_{14}N_{3}O_{2}/C_{7}H_{5}O_{2}/C_{1}H_{2}O, (I), and similar co-crystallization with 3,5-dinitrosalicylic acid yields the 2-carboxy-4,6-dinitrophenolate salt, C_{10}H_{14}N_{3}O_{2}/C_{7}H_{3}N_{2}O_{7}, (II). In the structure of (I), a combination of O—H···O, N—H···O and C—H···O hydrogen bonds links the components into sheets, while in the structure of (II), the supramolecular assembly, generated by hydrogen bonds of the same types as in (I), is three dimensional. Comparisons are made with the structures of some related compounds.

1. Chemical context

Piperazines and substituted piperazines are important pharmacophores, which can be found in many biologically active compounds (Berkheij, 2005) such as antifungal (Upadhyaya et al., 2004), anti-bacterial, anti-malarial and anti-psychotic agents (Chaudhary et al., 2006). Both the general pharmacological and specific antimicrobial activities of piperazine derivatives have been reviewed in recent years (Elliott, 2011; Kharb et al., 2012). Among specific examples of piperazine derivatives, N-(4-nitrophenyl)piperazine has found use in the control of potassium channels (Lu, 2007). The crystal structures of a number of 4-(4-nitrophenyl)piperazin-1-ium salts have been reported (Lu, 2007; Mahesha et al., 2022), and here we report the molecular and supramolecular structures of two further representatives of this family of salts, namely 4-(4-nitrophenyl)piperazin-1-ium benzoate monohydrate, C_{10}H_{14}N_{3}O_{2}/C_{7}H_{5}O_{2}/C_{1}H_{2}O, (I), and 4-(4-nitrophenyl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate, C_{10}H_{14}N_{3}O_{2}/C_{7}H_{3}N_{2}O_{7}, (II).

2. Structural commentary

In each of compounds (I) and (II) (Figs. 1 and 2), the piperazine ring adopts a chair conformation, with the ring-puckering angle $\theta$ (Cremer & Pople, 1975) calculated for the atom
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sequence (N11/C12/C13/N14/C15/C16) close to the ideal value of zero (Boeyens, 1978); \( \theta = 6.42 \) (11) for (I) and 8.75 (11)° for (II). However, in (I), the nitrophenyl substituent occupies an equatorial site, whereas in (II) this substituent occupies an axial site. In each compound, the \( N \)-nitrophenyl unit shows the pattern of distances typical of 4-nitroaniline derivatives, namely both \( C—N \) distances are short for their types (Allen et al., 1987), while the nitro \( N—O \) distances are long for their type. In addition, the distances C141 – C142 and C141 – C146 lie in the range 1.4049 (16) to 1.4132 (15) \( \text{Å} \) whereas the remaining \( C—C \) distances in the ring, which are in the range 1.3764 (17) to 1.3881 (15) \( \text{Å} \). These variations are most simply interpreted in terms of some 1,4-quinonoid type

\[ \text{C21—O21 distance, } \text{1.2788 (13) Å is more typical of those in ketones than those in phenols (Allen et al., 1987); the distances C21—C22 and C21—C26, 1.4394 (15) and 1.4340 (15) Å are longer than the remaining \( C—C \) distances in the ring, which are in the range 1.3747 (15) to 1.3869 (15). These observations, taken together, indicate that the negative charge in this anion is delocalized over atoms C22–C26 rather than being localized on atom O21 (see Scheme).} \]

In the anion of compound (II), the \( C21—O21 \) distance, 1.2788 (13) Å is more typical of those in ketones than those in phenols (Allen et al., 1987); the distances C21—C22 and C21—C26, 1.4394 (15) and 1.4340 (15) Å are longer than the remaining \( C—C \) distances in the ring, which are in the range 1.3747 (15) to 1.3869 (15). These observations, taken together, indicate that the negative charge in this anion is delocalized over atoms C22–C26 rather than being localized on atom O21 (see Scheme).

3. Supramolecular features

In each of compounds (I) and (II), the supramolecular assembly involves a combination of \( O—H···O, N—H···O \) and \( C—H···O \) hydrogen bonds, augmented in the case of (I) by a single \( C—H···π(\text{arene}) \) hydrogen bond: however, aromatic \( π···π \) stacking interactions are absent from both structures.

The supramolecular assembly in (I) is di-periodic and the formation of the sheet structure is readily analysed in terms of two mono-periodic sub-structures (Ferguson et al., 1998a; Gregson et al., 2000). Within the selected asymmetric unit for (I) (Fig. 1), the ionic components are linked by an asymmetric bifurcated (three-centre) \( N—H···(O,O) \) hydrogen bond (Table 1), while the water molecule is linked to the anion by an \( O—H···O \) hydrogen bond. In one of the two sub-structures, a combination of one two-centre \( N—H···O \) hydrogen bond and a second \( O—H···O \) hydrogen bond links these three-component aggregates (Fig. 1) into a chain of rings running parallel to the \([100]\) direction (Fig. 3) in which there are two different types of \( R_{2}^{1}(12) \) ring (Bernstein et al., 1995), centred at \((n, 0.5, 0.5)\) and \((n + 0.5, 0.5, 0.5)\), respectively, where \(n\) represents an integer in each case. The second sub-structure, which includes the \( C—H···O \) hydrogen bond (Table 1, Fig. 4), takes the form of another chain of rings in which \( R_{2}^{1}(12) \) rings centred at \((n + 0.5, n + 0.5, 0.5)\) alternate with \( R_{2}^{1}(10) \) rings

Table 1

| \( D—H···A \) | \( D—H \) | \( H···A \) | \( D···A \) | \( D—H···A \) |
|---------------|----------|----------|----------|----------|
| N11—H11···O271 | 0.926 (14) | 2.564 (14) | 3.1009 (13) | 117.4 (10) |
| N11—H11···O272 | 0.926 (14) | 1.857 (14) | 2.7781 (12) | 172.9 (13) |
| N11—H12···O31 | 0.920 (15) | 1.884 (15) | 2.7965 (14) | 171.0 (12) |
| O31—H31···O271 | 0.892 (18) | 1.757 (18) | 2.6486 (13) | 179 (3) |
| O31—H32···O272 | 0.908 (17) | 1.862 (17) | 2.7581 (12) | 168.8 (16) |
| C12—H12B···O272 | 0.99 | 2.45 | 3.3751 (15) | 156 |
| C146—H146···Cg1 | 0.95 | 2.67 | 3.4363 (13) | 138 |

Symmetry codes: (i) \(-x+1, -y+1, -z+1\); (ii) \(x, y, z\); (iii) \(-x, -y, -z+1\); (iv) \(-x+1, -y, -z+1\).
centred at \((n, n, 0.5)\), where \(n\) again represents an integer, so forming a chain of rings running parallel to the \([110]\) direction (Fig. 4). The combination of chains along \([100]\) and \([110]\) generates a sheet structure lying parallel to \((001)\). The single C—H/C1/C1/C1/C25 (arene) hydrogen bond (Table 1) lies within this sheet, and so has no influence on the dimensionality of the assembly.

The supramolecular assembly for compound (II), by contrast, is tri-periodic (three dimensional) and, as for (I), the formation of the framework is readily analysed in terms of simple sub-structures. Within the selected asymmetric unit (Fig. 2), there is an intramolecular O—H/C1/C1/C1 O hydrogen bond, and the hydroxyl H atom plays no part in the supramolecular assembly. The two independent components are linked by a very asymmetric bifurcated N—H/C1/C1/C1 (O,O) hydrogen bond (Table 2), and a two-centre N—H/C1/C1/C1 O hydrogen bond links these ion pairs into a chain of rings running parallel to the \([010]\) direction (Fig. 5). There are four C—H/C1/C1/C1 O hydrogen bonds in the structure of (II) and that involving atom C145 (Table 2) links the ion pairs into a second

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

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N11—H11·—O21 | 0.894 (14) | 1.869 (14) | 2.7356 (12) | 162.8 (13) |
| N11—H11·—O262 | 0.894 (14) | 2.396 (14) | 2.8937 (13) | 115.4 (11) |
| N11—H12·—O271 | 0.910 (14) | 1.874 (14) | 2.7668 (12) | 166.2 (12) |
| O272—H272·—O21 | 1.000 (17) | 1.549 (17) | 2.5020 (12) | 157.4 (15) |
| C12—H12·—O142w | 0.99 | 2.41 | 3.3921 (14) | 173 |
| C16—H16A·—O141u | 0.99 | 2.54 | 3.4906 (14) | 161 |
| C145—H145·—O2421i | 0.95 | 2.46 | 3.3927 (15) | 168 |
| C146—H146·—O241iv | 0.95 | 2.55 | 3.4227 (15) | 153 |

Symmetry codes: (i) \(-x+1, -y+1, -z+2\); (ii) \(-x+1, -y+1, -z+2\); (iii) \(-x+1, -y+1, -z+1\); (iv) \(-x+1, -y+1, -z+1\).

Figure 3
Part of the crystal structure of compound (I) showing the formation of a chain of hydrogen-bonded rings running parallel to the \([100]\) direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms bonded to C atoms have all been omitted.

Figure 4
Part of the crystal structure of compound (I) showing the formation of a chain of hydrogen-bonded rings running parallel to the \([110]\) direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms bonded to those C atoms that are not involved in the motif shown have been omitted.

Figure 5
Part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded chain of rings running parallel to \([010]\). Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, the H atoms bonded to C atoms have all been omitted.
The two C—H⋯O hydrogen bonds involving atoms C12 and C16 link inversion-related pairs of cations into a centrosymmetric motif containing $R_2^2(8)$ rings (Fig. 7), and the aggregates of this type are further linked by the final C—H⋯O hydrogen bond, that involves atom C146, to form a complex chain of rings running parallel to the [001] direction (Fig. 8). The combination of hydrogen-bonded chains parallel to [010], [001] and [101] generates a three-dimensional network. We also note a fairly short nitro–nitro contact, 2.823 (4) Å, between atom O142 at $(x, y, z)$ and atom N24 at $(1 + x, y, 1 + z)$: this probably represents a dipolar attraction between negatively charged O and positively charged N atoms.
4. Database survey

The first structure report on a salt of N-(4-nitrophenyl)-piperazine concerned the chloride salt, which crystallizes as a monohydrate (Lu, 2007); despite the presence of hydrogen bonds of N—H···O, N—H···Cl and O—H···Cl types, the supramolecular assembly is only mono-periodic. The structures of six salts of N-(4-nitrophenyl)piperazine with aromatic carboxylic acids have recently been reported (Mahesha et al., 2022); in all but one of these, the supramolecular assembly is mono-periodic, although it is di-periodic in the 4-ethoxybenzoate salt. This may be contrasted with the triperiodic assembly found here for compound (II).

In addition, we note that structures have been reported for a wide variety of salts derived from N-(4-fluorophenyl)-piperazine (Harish Chinthal, Yathirajan, Archana et al., 2020; Harish Chinthal, Yathirajan, Kavitha et al., 2020), and from N-(4-methoxyphenyl)piperazine (Kiran Kumar et al., 2019, 2020). Finally, the structure of 4-(2-methoxyphenyl)piperazin-1-ium 3,5-dinitrosalicylate has been reported, but without any description of discussion of the geometry of the anion (Subha et al., 2022).

5. Synthesis and crystallization

For the preparation of compounds (I) and (II), a solution of N-(4-nitrophenyl)piperazine (100 mg, 0.483 mmol) in methanol (10 ml) was mixed with a solution of either benzoic acid (59 mg, 0.483 mmol) for (I) or 3,5-dinitrosalicylic acid (110 mg, 0.483 mmol) for (II) in methanol/ethyl acetate (1:1 v/v, 20 ml). The solutions of the base and the corresponding acid were mixed, stirred at ambient temperature for 15 min, and then set aside to crystallize at ambient temperature and in the presence of air. After one week, crystals suitable for single-crystal X-ray diffraction were collected by filtration and dried in air: compound (I), pale yellow, m.p. 410–413 K; compound (II), orange, m.p. 446–448 K.

6. Refinement

Crystal data, data collection and refinement details are summarized in Table 3. All H atoms were located in difference maps. The H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions with C—H distances of 0.95 Å (aromatic) or 0.99 Å (CH2), and with \( U_{iso}(H) = 1.2U_{eq}(C) \). For the H atoms bonded to N or O atoms, the atomic coordinates were refined with \( U_{iso}(H) = 1.2U_{eq}(N) \) or \( 1.5U_{eq}(O) \), giving the N—H and O—H distances shown in Tables 1 and 2.

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Syntheses and crystal structures of 4-(4-nitrophenyl)piperazin-1-ium benzoate monohydrate and 4-(4-nitrophenyl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate

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Computing details
For both structures, data collection: APEX3 (Bruker, 2016); cell refinement: APEX3 (Bruker, 2016); data reduction: APEX3 (Bruker, 2016); program(s) used to solve structure: SHELXT2014/5 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015b); molecular graphics: PLATON (Spek, 2020); software used to prepare material for publication: SHELXL2014 (Sheldrick, 2015b) and PLATON (Spek, 2020).

4-(4-Nitrophenyl)piperazin-1-ium benzoate monohydrate (I)

Crystal data
C₁₀H₁₄N₃O₂⁺·C₇H₅O₂⁻·H₂O
Mr = 347.37
Triclinic, P\textoverline{1}
a = 6.0768 (3) Å
b = 7.4427 (4) Å
c = 18.4737 (9) Å
α = 78.894 (2)°
β = 85.870 (3)°
γ = 83.668 (2)°
V = 813.77 (7) Å³

Z = 2
F(000) = 368
D\textsubscript{c} = 1.418 Mg m\textsuperscript{-3}
Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 3737 reflections
θ\textsubscript{max} = 27.6°, θ\textsubscript{min} = 2.3°

Data collection
Bruker D8 Venture diffractometer
Radiation source: microsource
Multilayer mirror monochromator
ϕ and ω scans
Absorption correction: multi-scan
(SADABS; Krause \textit{et al.}, 2015)
T\textsubscript{min} = 0.912, T\textsubscript{max} = 0.971

27142 measured reflections
3737 independent reflections
3164 reflections with I > 2σ(I)

Refinement
Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.036
wR(F²) = 0.091
S = 1.04

3737 reflections
238 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed

SHELXL2014 (Sheldrick, 2015b) and PLATON (Spek, 2020).
H atoms treated by a mixture of independent and constrained refinement
\[ w = \frac{1}{\sigma^2(F_o^2) + (0.030P)^2 + 0.2902P} \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

\[(\Delta/\sigma)_{\text{max}} = 0.001\]
\[\Delta \rho_{\text{max}} = 0.25\ \text{e}\ \AA^{-3}\]
\[\Delta \rho_{\text{min}} = -0.20\ \text{e}\ \AA^{-3}\]

**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 (Å²)**

|   | x     | y     | z     | \(U_{iso}^*/U_{eq}\) |
|---|-------|-------|-------|----------------------|
| N11 | 0.22281 (16) | 0.29238 (14) | 0.44679 (5) | 0.0163 (2) |
| H11 | 0.172 (2) | 0.2234 (19) | 0.4908 (8) | 0.020* |
| H12 | 0.255 (2) | 0.403 (2) | 0.4565 (7) | 0.020* |
| C12 | 0.04640 (18) | 0.33021 (16) | 0.39257 (6) | 0.0175 (2) |
| H12A | -0.0854 | 0.3983 | 0.4129 | 0.021* |
| H12B | 0.0020 | 0.2124 | 0.3839 | 0.021* |
| C13 | 0.12736 (18) | 0.44242 (16) | 0.32002 (6) | 0.0172 (2) |
| H13A | 0.0112 | 0.4575 | 0.2838 | 0.021* |
| H13B | 0.1524 | 0.5664 | 0.3278 | 0.021* |
| N14 | 0.33332 (15) | 0.35614 (13) | 0.28973 (5) | 0.0147 (2) |
| C15 | 0.50502 (18) | 0.30670 (16) | 0.34411 (6) | 0.0163 (2) |
| H15A | 0.5561 | 0.4205 | 0.3546 | 0.020* |
| H15B | 0.6336 | 0.2365 | 0.3230 | 0.020* |
| C16 | 0.42143 (19) | 0.19258 (16) | 0.41561 (6) | 0.0177 (2) |
| H16A | 0.3830 | 0.0734 | 0.4063 | 0.021* |
| H16B | 0.5394 | 0.1671 | 0.4515 | 0.021* |
| C141 | 0.40488 (18) | 0.44038 (15) | 0.21853 (6) | 0.0149 (2) |
| C142 | 0.26572 (19) | 0.57118 (16) | 0.17307 (6) | 0.0199 (2) |
| H142 | 0.1188 | 0.6040 | 0.1906 | 0.024* |
| C143 | 0.3394 (2) | 0.65311 (16) | 0.10304 (6) | 0.0213 (3) |
| H143 | 0.2432 | 0.7399 | 0.0725 | 0.026* |
| C144 | 0.5535 (2) | 0.60750 (16) | 0.07820 (6) | 0.0189 (2) |
| C145 | 0.6944 (2) | 0.47632 (16) | 0.12051 (6) | 0.0199 (2) |
| H145 | 0.8406 | 0.4442 | 0.1021 | 0.024* |
| C146 | 0.61972 (19) | 0.39295 (16) | 0.18972 (6) | 0.0178 (2) |
| H146 | 0.7152 | 0.3015 | 0.2186 | 0.021* |
| N144 | 0.63672 (18) | 0.70469 (14) | 0.00703 (6) | 0.0242 (2) |
| O141 | 0.50758 (17) | 0.81136 (14) | -0.03222 (5) | 0.0352 (2) |
| O142 | 0.83458 (17) | 0.67621 (15) | -0.01004 (6) | 0.0421 (3) |
| C21 | 0.13555 (18) | 0.09842 (14) | 0.71032 (6) | 0.0156 (2) |
| C22 | -0.05414 (19) | 0.01147 (15) | 0.73666 (6) | 0.0170 (2) |
| H22 | -0.1565 | -0.0072 | 0.7030 | 0.020* |
| C23 | -0.0937 (2) | -0.04784 (16) | 0.81206 (6) | 0.0207 (2) |
| H23 | -0.2239 | -0.1058 | 0.8299 | 0.025* |
| C24 | 0.0564 (2) | -0.02246 (16) | 0.86119 (6) | 0.0229 (3) |
| Atomic displacement parameters (Å²) |
|-----------------------------------|
| **U**¹¹ | **U**¹² | **U**¹³ | **U**²¹ | **U**²² | **U**²³ |
|-----------------------------------|
| N11   | 0.0197 (5) | 0.0150 (5) | 0.0136 (5) | -0.0029 (4) | 0.0017 (4) | -0.0017 (4) |
| C12   | 0.0152 (5) | 0.0199 (6) | 0.0168 (5) | -0.0021 (4) | 0.0012 (4) | -0.0027 (4) |
| C13   | 0.0311 (6) | 0.0186 (5) | 0.0153 (5) | -0.0005 (4) | 0.0012 (4) | -0.0023 (4) |
| N14   | 0.0129 (4) | 0.0170 (5) | 0.0133 (4) | -0.0004 (4) | 0.0003 (3) | -0.0016 (3) |
| C15   | 0.0142 (5) | 0.0186 (5) | 0.0153 (5) | -0.0005 (4) | 0.0012 (4) | -0.0023 (4) |
| C16   | 0.0189 (6) | 0.0174 (5) | 0.0154 (5) | -0.0014 (4) | 0.0004 (4) | -0.0015 (4) |
| C141  | 0.0166 (5) | 0.0138 (5) | 0.0149 (5) | -0.0035 (4) | 0.0003 (4) | -0.0038 (4) |
| C142  | 0.0173 (6) | 0.0229 (6) | 0.0178 (6) | -0.0004 (5) | 0.0000 (4) | -0.0011 (4) |
| C143  | 0.0238 (6) | 0.0203 (6) | 0.0181 (6) | -0.0008 (5) | 0.0005 (5) | -0.0022 (4) |
| C144  | 0.0267 (6) | 0.0166 (5) | 0.0138 (5) | -0.0061 (5) | 0.0018 (4) | -0.0023 (4) |
| C145  | 0.0203 (6) | 0.0205 (6) | 0.0186 (5) | -0.0023 (5) | 0.0036 (4) | -0.0048 (4) |
| C146  | 0.0181 (6) | 0.0167 (5) | 0.0174 (5) | 0.0009 (4) | 0.0003 (4) | 0.0020 (4) |
| N144  | 0.0324 (6) | 0.0222 (5) | 0.0170 (5) | 0.0011 (5) | 0.0035 (4) | 0.0018 (4) |
| O141  | 0.0423 (6) | 0.0372 (6) | 0.0203 (5) | 0.0024 (5) | 0.0027 (4) | 0.0089 (4) |
| O142  | 0.0364 (6) | 0.0463 (6) | 0.0329 (5) | 0.0013 (5) | 0.0165 (4) | 0.0092 (5) |
| C21   | 0.0167 (5) | 0.0110 (5) | 0.0180 (5) | 0.0009 (4) | 0.0008 (4) | 0.0019 (4) |
| C22   | 0.0169 (5) | 0.0158 (5) | 0.0186 (5) | -0.0011 (4) | 0.0002 (4) | 0.0043 (4) |
| C23   | 0.0226 (6) | 0.0172 (6) | 0.0209 (6) | -0.0024 (5) | 0.0006 (5) | 0.0006 (5) |
| C24   | 0.0348 (7) | 0.0164 (6) | 0.0158 (5) | 0.0018 (5) | 0.0008 (5) | 0.0021 (4) |
| C25   | 0.0302 (7) | 0.0180 (6) | 0.0236 (6) | 0.0003 (5) | -0.0010 (5) | 0.0007 (5) |
| C26   | 0.0190 (6) | 0.0142 (5) | 0.0271 (6) | -0.0019 (4) | 0.0013 (5) | -0.0024 (4) |
| C27   | 0.0171 (5) | 0.0130 (5) | 0.0211 (6) | 0.0017 (4) | 0.0036 (4) | 0.0004 (4) |
| O271  | 0.0260 (5) | 0.0318 (5) | 0.0282 (5) | -0.0127 (4) | 0.0070 (4) | 0.0012 (4) |
| O272  | 0.0275 (5) | 0.0213 (4) | 0.0157 (4) | -0.0036 (3) | 0.0013 (3) | -0.0020 (3) |
| O31   | 0.0203 (4) | 0.0214 (4) | 0.0228 (4) | -0.0045 (4) | 0.0042 (3) | -0.0044 (3) |

**Geometric parameters (Å, °)**

| N11—C16 | 1.4857 (14) | C144—C145 | 1.3841 (17) |
| N11—C12 | 1.4870 (14) | C144—N144 | 1.4581 (14) |
| N11—H11 | 0.925 (14) | C145—C146 | 1.3780 (16) |
| N11—H12 | 0.921 (15) | C145—H145 | 0.9500 |
| C12—C13 | 1.5167 (15) | C146—H146 | 0.9500 |
| Bond          | Distance (Å) | Bond          | Distance (Å) | Distance (Å) |
|---------------|--------------|---------------|--------------|--------------|
| C12—H12A      | 0.9900       | N144—O141     | 1.2226 (14)  |
| C12—H12B      | 0.9900       | N144—O142     | 1.2268 (14)  |
| C13—N14       | 1.4671 (14)  | C21—C26       | 1.3911 (16)  |
| C13—H13A      | 0.9900       | C21—C22       | 1.3953 (16)  |
| C13—H13B      | 0.9900       | C21—C27       | 1.5084 (15)  |
| N14—C141      | 1.4044 (14)  | C22—C23       | 1.3896 (16)  |
| N14—C15       | 1.4688 (14)  | C22—H22       | 0.9500       |
| C15—C16       | 1.5134 (15)  | C23—C24       | 1.3840 (18)  |
| C15—H15A      | 0.9900       | C23—H23       | 0.9500       |
| C15—H15B      | 0.9900       | C24—C25       | 1.3869 (18)  |
| C16—H16A      | 0.9900       | C24—H24       | 0.9500       |
| C16—H16B      | 0.9900       | C25—C26       | 1.3838 (17)  |
| C141—C142     | 1.4049 (16)  | C25—H25       | 0.9500       |
| C141—C146     | 1.4081 (16)  | C26—H26       | 0.9500       |
| C142—C143     | 1.3853 (16)  | C27—O271      | 1.2476 (14)  |
| C142—H142     | 0.9500       | C27—O272      | 1.2692 (14)  |
| C143—C144     | 1.3764 (17)  | O31—H31       | 0.893 (17)   |
| C143—H143     | 0.9500       | O31—H32       | 0.909 (17)   |
| C16—N11—C12   | 109.33 (9)   | C141—C142—H142| 119.5        |
| C16—N11—H11   | 110.2 (8)    | C144—C143—C142| 119.36 (11)  |
| C12—N11—H11   | 110.3 (8)    | C144—C143—H143| 120.3        |
| C16—N11—H12   | 111.3 (8)    | C142—C143—H143| 120.3        |
| C12—N11—H12   | 108.0 (8)    | C143—C144—C145| 121.48 (11)  |
| H11—N11—H12   | 107.6 (12)   | C143—C144—N144| 119.32 (11)  |
| N11—C12—C13   | 110.80 (9)   | C145—C144—N144| 119.16 (11)  |
| N11—C12—H12A  | 109.5        | C146—C145—C144| 119.05 (11)  |
| C13—C12—H12A  | 109.5        | C146—C145—H145| 120.5        |
| N11—C12—H12B  | 109.5        | C144—C145—H145| 120.5        |
| C13—C12—H12B  | 109.5        | C145—C146—C141| 121.42 (10)  |
| H12A—C12—H12B | 108.1        | C145—C146—H146| 119.3        |
| N14—C13—C12   | 112.32 (9)   | C141—C146—H146| 119.3        |
| N14—C13—H13A  | 109.1        | O141—N144—O142| 123.28 (11)  |
| C12—C13—H13A  | 109.1        | O141—N144—C144| 118.78 (11)  |
| N14—C13—H13B  | 109.1        | O142—N144—C144| 117.94 (10)  |
| C12—C13—H13B  | 109.1        | C26—C21—C22   | 119.26 (10)  |
| H13A—C13—H13B | 107.9        | C26—C21—C27   | 119.54 (10)  |
| C141—N14—C13  | 115.67 (9)   | C22—C21—C27   | 121.19 (10)  |
| C141—N14—C15  | 115.77 (9)   | C23—C22—C21   | 120.20 (11)  |
| C13—N14—C15   | 112.34 (9)   | C23—C22—H22   | 119.9        |
| N14—C15—C16   | 112.12 (9)   | C21—C22—H22   | 119.9        |
| N14—C15—H15A  | 109.2        | C24—C23—C22   | 119.98 (11)  |
| C16—C15—H15A  | 109.2        | C24—C23—H23   | 120.0        |
| N14—C15—H15B  | 109.2        | C22—C23—H23   | 120.0        |
| C16—C15—H15B  | 109.2        | C23—C24—C25   | 120.06 (11)  |
| H15A—C15—H15B | 107.9        | C23—C24—H24   | 120.0        |
| N11—C16—C15   | 110.12 (9)   | C25—C24—H24   | 120.0        |
| N11—C16—H16A  | 109.6        | C26—C25—C24   | 120.10 (11)  |
C15—C16—H16A 109.6  C26—C25—H25 119.9
N11—C16—H16B 109.6  C24—C25—H25 119.9
C15—C16—H16B 109.6  C25—C26—C21 120.38 (11)
H16A—C16—H16B 108.2  C25—C26—H26 119.8
N14—C141—C142 121.85 (10) C24—C25—H25 119.8
N14—C141—C146 120.53 (10) O271—C27—O272 124.07 (11)
C142—C141—C146 117.62 (10) O271—C27—C21 117.57 (11)
C143—C142—C141 121.01 (11) O272—C27—C21 118.35 (10)
C143—C142—H142 119.5  H31—O31—H32 106.4 (14)

C16—N11—C12—C13 −58.34 (12)  C144—C145—C146—C141 −0.95 (17)
N11—C12—C13—N14 54.53 (12)  N14—C141—C146—C145 −178.85 (10)
C12—C13—N14—C141 172.83 (9)  C142—C141—C146—C145 2.33 (17)
C12—C13—N14—C146 51.20 (12)  C143—C141—C146—C145 −178.85 (10)
C13—N14—C141—C142 −13.70 (15)  C142—C141—C146—C145 2.33 (17)
C13—N14—C141—C146 167.53 (10)  C143—C141—C146—C145 −178.85 (10)
C12—N11—C16—C15 59.27 (12)  C145—C144—N144—O141 −7.29 (17)
N11—C16—C15—N14 −56.67 (12)  C145—C144—N144—O142 175.18 (11)
N11—C16—C15—C16 52.40 (12)  C145—C144—N144—O142 −5.55 (17)
N11—C16—C15—C16 −171.67 (9)  C26—C21—C22—C23 0.09 (16)
C12—N11—C16—C15 59.27 (12)  C27—C21—C22—C23 −179.53 (10)
N11—C16—C15—C16 −148.15 (11)  C22—C23—C24—C25 −0.69 (17)
N11—C16—C15—C16 172.83 (9)  C22—C23—C24—C25 0.35 (18)
C12—N11—C16—C15 −148.15 (11)  C23—C24—C25—C26 0.59 (18)
C12—N11—C16—C15 172.83 (9)  C24—C25—C26—C21 −1.19 (17)
C141—C142—C143—C144 −1.33 (17)  C22—C21—C26—C25 −179.52 (10)
C141—C142—C143—C144 1.02 (18)  C22—C21—C26—C25 0.85 (17)
C142—C143—C144—C145 −2.49 (18)  C22—C21—C26—C25 −179.52 (10)
C142—C143—C144—N144 −174.99 (11)  C22—C21—C26—C25 −179.52 (10)
C143—C144—C145—C146 −1.51 (18)  C22—C21—C26—C25 −179.52 (10)
N144—C144—C145—C146 175.97 (10)  C22—C21—C26—C25 −179.52 (10)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C21–C26 ring.

| D—H···A  | D—H  | H···A  | D···A  | D—H···A |
|---------|------|-------|-------|--------|
| N11·H11···O271 | 0.926 (14) | 2.564 (14) | 3.1009 (13) | 117.4 (10) |
| N11·H11···O272 | 0.926 (14) | 1.857 (14) | 2.7781 (12) | 172.9 (13) |
| N11·H12···O31i | 0.920 (15) | 1.884 (15) | 2.7965 (14) | 171.0 (12) |
| O31·H31···O271 | 0.892 (18) | 1.757 (18) | 2.6486 (13) | 179 (3) |
| O31·H32···O272ii | 0.908 (17) | 1.862 (17) | 2.7581 (12) | 168.8 (16) |
| C12·H12B···O272iii | 0.99 | 2.45 | 3.3751 (15) | 156 |
| C146·H146···Cg1iv | 0.95 | 2.67 | 3.4363 (13) | 138 |

Symmetry codes: (i) −x+1, −y+1, −z+1; (ii) x+1, y, z; (iii) −x, −y, −z+1; (iv) −x+1, −y, −z+1.
4-(4-Nitrophenyl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate (II)

Crystal data

C\textsubscript{10}H\textsubscript{14}N\textsubscript{3}O\textsubscript{2}+·C\textsubscript{7}H\textsubscript{3}N\textsubscript{2}O\textsubscript{7}−

Mr = 435.35

Triclinic, \(P\bar{1}\)

\(a = 7.9599 (4) \text{ Å}\)

\(b = 8.5391 (4) \text{ Å}\)

\(c = 14.2227 (5) \text{ Å}\)

\(\alpha = 90.426 (2)°\)

\(\beta = 105.273 (1)°\)

\(\gamma = 98.538 (2)°\)

\(V = 921.15 (7) \text{ Å}^3\)

\(Z = 2\)

\(F(000) = 452\)

\(D_c = 1.570 \text{ Mg m}^{-3}\)

Mo K\(\alpha\) radiation, \(\lambda = 0.71073 \text{ Å}\)

Cell parameters from 4212 reflections

\(\theta = 2.4–27.5°\)

\(\mu = 0.13 \text{ mm}^{-1}\)

\(T = 90 \text{ K}\)

Block, orange

\(V = 921.15 (7) \text{ Å}^3\)

0.22 × 0.18 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer

Radiation source: microsource

Multilayer mirror monochromator

\(\phi\) and \(\omega\) scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

\(T_{\text{min}} = 0.919, T_{\text{max}} = 0.971\)

38287 measured reflections

4212 independent reflections

3662 reflections with \(I > 2\sigma(I)\)

\(R_{\text{int}} = 0.043\)

\(\theta_{\text{max}} = 27.5°, \theta_{\text{min}} = 2.4°\)

\(h = -10→10\)

\(k = -11→11\)

\(l = -18→17\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.029\)

\(wR(F^2) = 0.077\)

\(S = 1.04\)

4212 reflections

289 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: mixed

\(H\) atoms treated by a mixture of independent and constrained refinement

\(w = 1/[\sigma^2(F_c^2) + (0.029P)^2 + 0.3527P]\)

where \(P = (F_c^2 + 2F_e^2)/3\)

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

\(\Delta\rho_{\text{max}} = 0.28 \text{ e Å}^{-3}\)

\(\Delta\rho_{\text{min}} = -0.18 \text{ e Å}^{-3}\)

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 (Å\(^2\))

|     | \(x\)        | \(y\)        | \(z\)        | \(U_{eq}\) |
|-----|--------------|--------------|--------------|-----------|
| N11 | 0.30883 (12) | 0.10386 (11) | 0.64405 (7)  | 0.01693 (19) |
| H11 | 0.2967 (17)  | 0.1840 (17)  | 0.6040 (10)  | 0.020*    |
| H12 | 0.2796 (17)  | 0.0129 (17)  | 0.6057 (10)  | 0.020*    |
| C12 | 0.17915 (14) | 0.10314 (13) | 0.70340 (8)  | 0.0188 (2) |
| H12A| 0.0579       | 0.0733       | 0.6608       | 0.023*    |
| H12B| 0.1890       | 0.2107       | 0.7328       | 0.023*    |
| C13 | 0.21403 (14) | -0.01389 (13)| 0.78350 (8)  | 0.0187 (2) |
| H13A| 0.1321       | -0.0092      | 0.8248       | 0.022*    |
| H13B| 0.1931       | -0.1229      | 0.7541       | 0.022*    |

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### Atomic displacement parameters (Å²)

|   | $U_{11}$  | $U_{22}$  | $U_{33}$  | $U_{12}$  | $U_{13}$  | $U_{23}$  |
|---|-----------|-----------|-----------|-----------|-----------|-----------|
| N11 | 0.0230 (5) | 0.0128 (4) | 0.0146 (4) | 0.0040 (4) | 0.0037 (4) | −0.0003 (4) |
| C12 | 0.0177 (5) | 0.0181 (5) | 0.0198 (5) | 0.0037 (4) | 0.0032 (4) | −0.0033 (4) |
| C13 | 0.0188 (5) | 0.0191 (5) | 0.0171 (5) | −0.0008 (4) | 0.0051 (4) | −0.0030 (4) |
| N14 | 0.0184 (4) | 0.0179 (4) | 0.0142 (4) | 0.0015 (4) | 0.0045 (3) | −0.0018 (3) |
| C15 | 0.0195 (5) | 0.0162 (5) | 0.0159 (5) | 0.0045 (4) | 0.0048 (4) | −0.0012 (4) |

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*Acta Cryst. (2022), E78, 840-845*
### Geometric parameters (Å, °)

| Bond/Distance | Value          |
|---------------|----------------|
| N11—C16       | 1.4919 (14)    |
| N11—C12       | 1.4951 (14)    |
| N11—H11       | 0.894 (14)     |
| N11—H12       | 0.910 (14)     |
| C12—C13       | 1.5187 (15)    |
| C12—H12A      | 0.9900         |
| C12—H12B      | 0.9900         |
| C13—N14       | 1.4635 (14)    |
| C13—H13A      | 0.9900         |
| C13—H13B      | 0.9900         |
| N14—C141      | 1.3812 (13)    |
| N14—C15       | 1.4625 (14)    |
| C15—C16       | 1.5169 (15)    |
| C15—H15A      | 0.9900         |
| C15—H15B      | 0.9900         |
| C16—H16A      | 0.9900         |
| C16—H16B      | 0.9900         |
| C141—C142     | 1.4125 (15)    |
| C141—C146     | 1.4133 (15)    |

### Supporting Information

Geometric parameters (Å, °) for the crystal structure of [insert structure details here].
C142—C143 1.3774 (15)  
C142—H142 0.9500  
C143—C144 1.3881 (15)  
C143—H143 0.9500  
C144—C145 1.3875 (15)  
C144—N144 1.4437 (13)  
C16—N11—C12 113.90 (8)  
C16—N11—H11 108.4 (9)  
C12—N11—H11 108.3 (9)  
C16—N11—H12 111.0 (8)  
C12—N11—H12 108.3 (9)  
H11—N11—H12 106.8 (12)  
N11—C12—C13 110.16 (9)  
N11—C12—H12A 109.6  
C13—C12—H12A 109.6  
N11—C12—H12B 109.6  
C13—C12—H12B 109.6  
H12A—C12—H12B 108.1  
N14—C13—C12 109.99 (9)  
N14—C13—H13A 109.7  
C12—C13—H13A 109.7  
N14—C13—H13B 109.7  
C12—C13—H13B 109.7  
H13A—C13—H13B 108.2  
C141—N14—C13 121.02 (9)  
C141—N14—C14 121.10 (9)  
C15—N14—C13 109.06 (8)  
N14—C15—C16 110.28 (9)  
N14—C15—H16A 109.7  
C15—C16—H16A 109.7  
N11—C16—C15 109.87 (9)  
N11—C16—H16A 109.7  
C15—C16—H16A 109.7  
N11—C16—H16B 109.7  
C15—C16—H16B 109.7  
N12—C17—C18 121.47 (10)  
N12—C17—H17A 109.2  
C18—C17—H17A 109.2  
C141—C142—C143 117.30 (10)  
C141—C142—H142 119.4  
C142—C143—C144 119.59 (10)  
C142—C143—H143 120.2  
O272—H272 1.003 (18)  
N24—O242 1.2277 (12)  
N24—O241 1.2389 (12)  
N26—O262 1.2303 (12)  
N26—O261 1.2343 (12)  
C144—C143—H143 120.2  
C145—C144—C143 120.87 (10)  
C145—C144—N144 119.48 (10)  
C143—C144—N144 119.67 (10)  
C146—C145—C144 121.42 (10)  
C146—C145—H145 120.3  
C144—C145—H145 120.3  
C145—C146—C141 121.48 (10)  
C145—C146—H146 119.3  
C141—C146—H146 119.3  
C21—C22—C26 124.86 (10)  
C22—C23—C24 120.26 (10)  
C22—C23—H23 119.9  
C23—C24—C23 117.95 (9)  
C23—C24—N24 117.95 (9)  
C25—C24—N24 117.95 (9)  
C26—C25—H25 120.5  
C26—C25—C24 120.5  
C26—C25—N26 115.98 (9)  
C25—C26—N26 120.55 (9)  
C27—O272—H272 105.0 (10)  
O242—N24—O241 123.40 (9)  
O242—N24—C24 118.92 (9)  
O241—N24—C24 117.67 (9)  
O262—N26—O261 122.85 (9)  
O262—N26—C26 119.17 (9)  
O261—N26—C26 117.95 (9)
C16—N11—C12—C13 −50.40 (12) C26—C21—C22—C23 −1.93 (15)
N11—C12—C13—N14 56.08 (11) C26—C21—C22—C27 −179.98 (10)
C12—C13—N14—C15 −63.61 (11) C21—C22—C23—C24 2.55 (16)
C141—N14—C15—C16 −83.73 (12) C27—C22—C23—C24 −179.35 (10)
C13—N14—C15—C16 63.98 (11) C22—C23—C24—C25 −0.39 (16)
C12—N11—C16—C15 50.41 (11) C22—C23—C24—N24 −178.48 (9)
N14—C15—C16—N11 −56.47 (11) C23—C24—C25—C26 −2.25 (16)
C13—N14—C141—C142 164.67 (10) N24—C24—C25—C26 175.84 (9)
C141—C142—C143—C144 0.36 (16) C24—C25—C26—N26 −175.67 (9)
C142—C143—C144—C145 −179.67 (10) C23—C22—C27—O271 5.49 (16)
C142—C143—C144—N144 −2.02 (16) C23—C22—C27—O272 −174.21 (10)
C143—C144—N144—O142 1.91 (15) N144—C144—C145—C146 −179.86 (10)
C145—C144—N144—O141 4.37 (15) C141—C142—C143—C144 −176.09 (10)
C142—C141—C146—C145 2.09 (16) C141—C142—C143—C144 176.14 (10)
C145—C141—C146—C145 2.09 (16) C141—C142—C143—C144 161.38 (10)
C145—C144—N144—O142 1.91 (15) C144—C145—C146—C141 −176.08 (10)
C145—C144—N144—O141 −175.95 (10) C145—C146—C141—C142 −176.08 (10)
O21—C21—C22—C23 179.56 (10) C25—C26—N26—O262 150.18 (10)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|-------|-------|--------|
| N11—H11···O21 | 0.894 (14) | 1.869 (14) | 2.7356 (12) | 162.8 (13) |
| N11—H11···O262 | 0.894 (14) | 2.396 (14) | 2.8937 (13) | 115.4 (11) |
| N11—H12···O271ii | 0.910 (14) | 1.874 (14) | 2.7668 (12) | 166.2 (12) |
| O272—H272···O21i | 1.000 (17) | 1.549 (17) | 2.5020 (12) | 157.4 (15) |
| C12—H12···O142ii | 0.99 | 2.41 | 3.3921 (14) | 173 |
| C16—H16···O141ii | 0.99 | 2.54 | 3.4906 (14) | 161 |
| C145—H145···O242iii | 0.95 | 2.46 | 3.3927 (15) | 168 |
| C146—H146···O241iv | 0.95 | 2.55 | 3.4227 (15) | 153 |

Symmetry codes: (i) x, y−1, z; (ii) −x+1, −y+1, −z+2; (iii) x+1, y, z+1; (iv) −x+1, −y+1, −z+1.