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# A resonance-assisted intramolecular hydrogen bond in compounds containing 2-hydroxy-3,5-dinitrobenzoic acid and its various deprotonated forms: redetermination of several related structures

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**CCDC references**: 1063245; 1862187; 1862188; 1862189; 1862190; 1862191; 1862192; 1862193; 1862194; 1862195; 1862196; 1862197; 1862198

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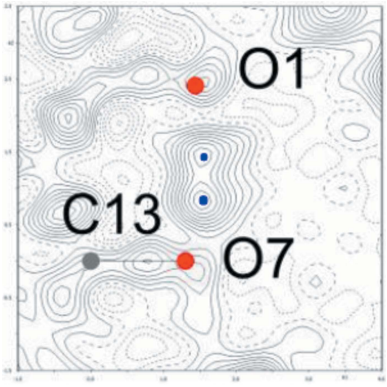
A large number of structural determinations of compounds containing 2-hydroxy-3,5-dinitrobenzoic acid (**I**) and its various deprotonated forms, 2-hydroxy-3,5-dinitrobenzoate (**II**) or 2-carboxy-4,6-dinitrophenolate (**III**), are biased. The reason for the bias follows from incorrectly applied constraints or restraints on the *bridging hydrogen*, which is involved in the intramolecular hydrogen bond between the neighbouring carboxylic/carboxylate and oxo/hydroxy groups. This hydrogen bond belongs to the category of resonance-assisted hydrogen bonds. The present article suggests corrections for the following structure determinations that have been published in *Acta Crystallographica*: DUJZAK, JEVNAA, LUDFUL, NUQVEB, QIQJAD, SAFGUD, SEDKET, TIYZIM, TUJPEV, VABZIJ, WADXOR, YAXPOE [refcodes are taken from the Cambridge Structural Database [CSD; Groom *et al.* (2016). *Acta Cryst.* B**72**, 171–179]. The structural features of the title molecules in all the retrieved structures, together with structures that contain 3,5-dinitro-2-oxidobenzoate (**IV**), are discussed. Attention is paid to the localization of the above-mentioned *bridging hydrogen*, which can be situated closer to the O atom of the carboxylate/carboxylic group or that of the hydroxy/oxo group. In some cases, it is disordered between the two O atoms. The position of the *bridging hydrogen* seems to be dependent on the p$K_a$(base) although with exceptions. A stronger basicity enhances the probability of the presence of a phenolate (**III**). The present article examines the problem of the refinement of such a *bridging hydrogen* as well as that of the hydrogen atoms involved in the hydroxy and primary and secondary amine groups. It appears that the best model, in many cases, is obtained by fixing the hydrogen-atom position found in the difference electron-density map while refining its isotropic displacement parameter.

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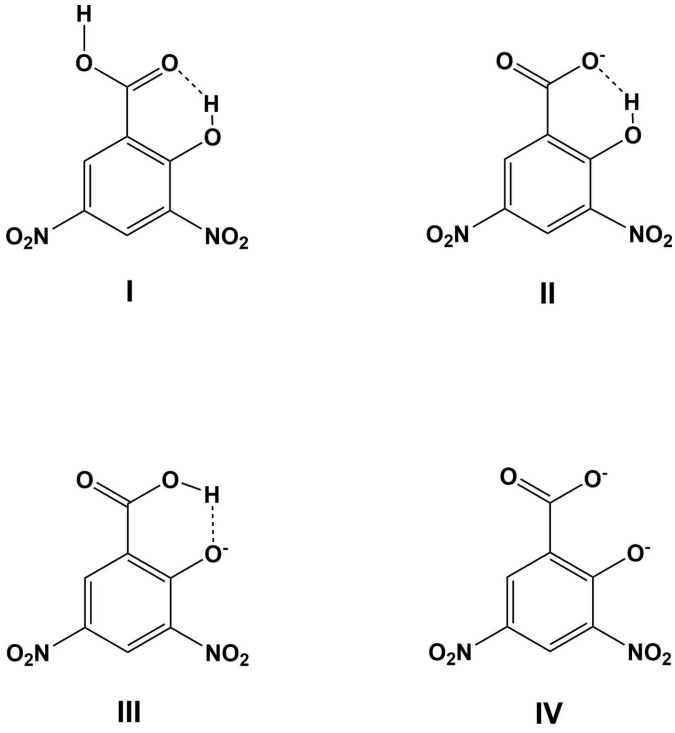
## 1. Chemical context

2-Hydroxy-3,5-dinitrobenzoic acid (**I**; alternatively 3,5-dinitrosalicylic acid, DNSA), 2-hydroxy-3,5-dinitrobenzoate (**II**; alternatively 3,5-dinitrosalicylate), 2-carboxy-4,6-dinitrophenolate (**III**) and 3,5-dinitro-2-oxidobenzoate (**IV**), are molecules that have interesting structural and chemical features. Such molecules have been studied because of the proton transfer from the carboxylic group, which is dependent on its environment (*e.g.* Smith *et al.*, 2007). Thus, three deprotonated forms of molecule **I** have been observed. The last one, **IV**, is deprived of all of the hydrogen atoms while the others differ in the localization of the hydrogen atom involved in the intramolecular hydrogen bond between the O atoms of the carboxylate/carboxylic and the hydroxy/oxo groups. In the different structures, this hydrogen atom may be closer to

either oxygen atom, depending on the properties of each particular structure. In some cases, this hydrogen atom may even be disordered. In the following, it will be referred to as a *bridging hydrogen*.




Such a *bridging hydrogen* is a part of a resonance-assisted moiety (Gilli & Gilli, 2009) composed of six atoms with the pertinent bonds being *D*1, *D*2, *D*3, *D*4, *D*11 and *D*12, as shown in Fig. 1*a*. However, the delocalized bonds can be further extended within the molecule, especially to the C═O/C—OH bond (*D*1/*D*5 in Fig. 1*a*). Resonance-assisted hydrogen bonds tend to be stronger and therefore the *bridging hydrogen* should be displaced towards the hydrogen-bond centre. On the other hand, O···H···O hydrogen bonds with a *bridging hydrogen* that is situated about its centre are usually observed for strong intramolecular hydrogen bonds with the O···O distances being shorter than 2.5 Å (Gilli & Gilli, 2009), while the O···H···O angles tend to be close to 180° (Jeffrey, 1995). The $O_{carboxylate/carboxylicgroup}\cdots O_{hydroxy/oxo\ group}$ distance can be as short as 2.41 Å in some 2-hydroxy-3,5-dinitrobenzoates (**II**) or 2-carboxy-4,6-dinitrophenolates (**III**); however, the O···H···O angle, which is *ca* 160°, situates it in a category of its own.

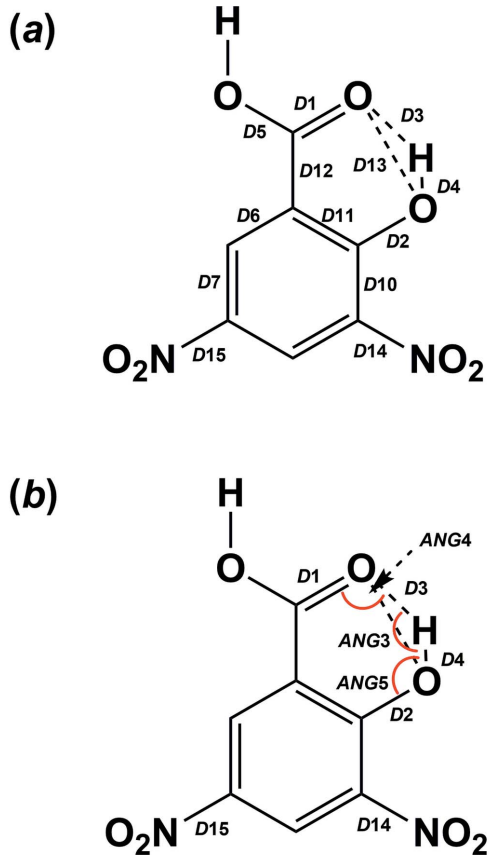
The above-mentioned features of the intramolecular O···H···O hydrogen bond in the molecules considered herein have been ignored on many occasions by incorrectly applied constraints or severe restraints on the O—H distances, 0.82 or 0.84 Å, together with angle constraints/restraints equal to 109° as proposed by *SHELXL* (Sheldrick, 2008, 2015).

A robust indication whether the *bridging hydrogen* has been positioned correctly follows from the bond distances C═O/C—O of the involved carboxylate/carboxylic and hydroxyl/oxo groups, although there are a few exceptions in which the *bridging hydrogen* is attached to the oxygen forming a slightly shorter C—O distance. These exceptions will be mentioned briefly below. Thus, it seems that a considerable number of the structures containing the molecules **I**–**IV** could have been determined more correctly with a more realistic description of the pertinent hydrogen bond in these molecular fragments.

A search of the Cambridge Structural Database (CSD, Version 3.58, last update May 2017; Groom *et al.*, 2016) indicated that 27 structures out of 53 reported as 2-hydroxy-3,5-dinitrobenzoates (**II**) seem to be suspect; 21 structures out of 70 reported as 2-carboxy-4,6-dinitrophenolates (**III**) seem to be suspect, and nine structures out of 15 that contain a molecule of 2-hydroxy-3,5-dinitrobenzoic acid (**I**) also appear to be suspect. Figs. 2*a* and 2*b* illustrate this situation for 2-hydroxy-3,5-dinitrobenzoates (**II**) and 2-carboxy-4,6-dinitrophenolates (**III**), respectively.

It is plausible to expect that the environment affects the position of the *bridging hydrogen*. Therefore, it can be assumed that the proton transfer stemming from the carboxyl group will affect its position.

The data for the suspect structures published in *Acta Crystallographica* were retrieved from the journal's web page




***ANG*1 - dihedral angle between plane of $NO_2$ group (*D*14) and the benzene ring**
***ANG*2 - dihedral angle between plane of $NO_2$ group (*D*15) and the benzene ring**

**Figure 1**
Definition of bonds and various angles in **I**–**IV**.

**Table 1**
Experimental details.

| | DUJZAK | JEVNAA | LUDFUL | NUQVEB |
|---|---|---|---|---|
| Crystal data | | | | |
| Chemical formula | $[Ag(C_9H_7NO)_2](C_7H_3N_2O_7)$ | $[Zn(C_3H_4N_2)_4](C_7H_3N_2O_7)_2$ | $C_7H_4N_2O_7\cdot C_{12}H_8N_2$ | $C_6H_9N_2^+\cdot C_7H_3N_2O_7^-$ |
| $M_r$ | 625.30 | 791.93 | 408.33 | 336.27 |
| Crystal system, space group | Monoclinic, $P2_1$ | Monoclinic, $C2/c$ | Monoclinic, $P2_1/a$ | Triclinic, $P\overline{1}$ |
| Temperature (K) | 293 | 293 | 293 | 100 |
| $a, b, c$ (Å) | 9.0154 (18), 7.6122 (15), 17.138 (3) | 25.0809 (15), 6.7251 (4), 18.9145 (10) | 14.8002 (15), 7.4029 (16), 16.0091 (16) | 5.8673 (7), 8.0991 (9), 15.2437 (17) |
| $\alpha, \beta, \gamma$ (°) | 90, 104.38 (3), 90 | 90, 97.658 (6), 90 | 90, 96.395 (8), 90 | 86.844 (3), 84.252 (3), 81.209 (3) |
| $V$ (Å$^3$) | 1139.3 (4) | 3161.9 (3) | 1743.1 (5) | 711.69 (14) |
| $Z$ | 2 | 4 | 4 | 2 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.95 | 0.87 | 0.12 | 0.13 |
| Crystal size (mm) | 0.20 × 0.15 × 0.11 | 0.20 × 0.18 × 0.10 | 0.36 × 0.34 × 0.26 | 0.29 × 0.14 × 0.08 |
| Data collection | | | | |
| Diffractometer | Bruker SMART CCD area-detector | Bruker APEXII area-detector | Enraf–Nonius CAD-4 | Bruker APEX DUO CCD area-detector |
| Absorption correction | – | Multi-scan (*SADABS*; Bruker, 1999) | – | Multi-scan (*SADABS*; Bruker, 2009) |
| $T_{min}, T_{max}$ | – | 0.846, 0.918 | – | 0.963, 0.990 |
| No. of measured, independent and observed $[I > 3\sigma(I)]$ reflections | 10841, 4602, 4225 | 20634, 3635, 2152 | 8396, 4202, 1587 | 12709, 4943, 3677 |
| $R_{int}$ | 0.022 | 0.058 | 0.056 | 0.023 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.651 | 0.651 | 0.661 | 0.756 |
| Refinement | | | | |
| $R$ factors and goodness of fit | $R[F > 3\sigma(F)] = 0.023$, $wR(F) = 0.053$, $S = 1.34$ | $R[F > 3\sigma(F)] = 0.036$, $wR(F) = 0.075$, $S = 1.23$ | $R[F > 3\sigma(F)] = 0.044$, $wR(F) = 0.083$, $S = 1.08$ | $R[F > 3\sigma(F)] = 0.042$, $wR(F) = 0.109$, $S = 2.06$ |
| No. of reflections | 4602 | 3635 | 4202 | 4943 |
| No. of parameters | 356 | 244 | 274 | 222 |
| No. of restraints | 0 | 0 | 0 | 0 |
| 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 | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}, \Delta\rho_{min}$ (e Å$^{-3}$) | 0.44, −0.30 | 0.23, −0.23 | 0.29, −0.31 | 0.40, −0.32 |
| Absolute structure | 1800 of Friedel pairs used in the refinement | – | – | – |
| Absolute structure parameter | 0.004 (17) | – | – | – |

| | QIQJAD | SAFGUD | SEDKET | TIYZIM |
|---|---|---|---|---|
| Crystal data | | | | |
| Chemical formula | $C_9H_8Cl_2N_5^+\cdot C_7H_3N_2O_7^-\cdot\cdot C_3H_7NO$ | $[Ag(C_{12}H_6N_2O_2)](C_7H_3N_2O_7)$ | $C_5H_9N_2^+\cdot C_7H_3N_2O_7^-$ | $C_6H_{12}N_3^+\cdot C_7H_3N_2O_7^-$ |
| $M_r$ | 557.31 | 755.36 | 324.26 | 353.30 |
| Crystal system, space group | Triclinic, $P\overline{1}$ | Monoclinic, $P2_1/c$ | Monoclinic, $P2_1$ | Triclinic, $P\overline{1}$ |
| Temperature (K) | 294 | 174 | 293 | 173 |
| $a, b, c$ (Å) | 10.0227 (5), 10.5507 (5), 12.5359 (6) | 11.757 (2), 18.297 (4), 13.223 (3) | 8.1183 (7), 6.0636 (5), 14.1453 (11) | 7.0109 (4), 10.6617 (8), 10.7454 (7) |
| $\alpha, \beta, \gamma$ (°) | 81.858 (1), 71.888 (1), 70.009 (1) | 90, 103.91 (3), 90 | 90, 91.904 (1), 90 | 93.075 (6), 95.863 (5), 104.944 (6) |
| $V$ (Å$^3$) | 1183.1 (1) | 2761.1 (11) | 695.93 (10) | 769.30 (9) |
| $Z$ | 2 | 4 | 2 | 2 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ | Mo $K\alpha$ | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.34 | 0.81 | 0.13 | 1.09 |
| Crystal size (mm) | 0.16 × 0.14 × 0.08 | 0.3 × 0.24 × 0.2 | 0.40 × 0.27 × 0.11 | 0.22 × 0.14 × 0.12 |
| Data collection | | | | |
| Diffractometer | Bruker SMART APEX CCD area-detector | Oxford Diffraction Gemini R Ultra | Bruker SMART CCD | Agilent Xcalibur (Eos, Gemini) |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2001) | Multi-scan (*SADABS*; Bruker, 2002) | Multi-scan (*SADABS*; Bruker, 2002) | Multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012) |
| $T_{min}, T_{max}$ | 0.93, 0.97 | 0.780, 0.910 | 0.959, 0.986 | 0.925, 1.000 |
| No. of measured, independent and observed $[I > 3\sigma(I)]$ reflections | 13936, 5507, 4441 | 12726, 5013, 3100 | 3523, 2301, 1444 | 4664, 2953, 2426 |

**Table 1 (continued)**

| | QIQJAD | SAFGUD | SEDKET | TIYZIM |
|---|---|---|---|---|
| $R_{int}$ | 0.019 | 0.052 | 0.040 | 0.026 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.661 | 0.603 | 0.595 | 0.618 |
| Refinement | | | | |
| *R* factors and goodness of fit | $R[F > 3\sigma(F)] = 0.056$, $wR(F) = 0.147$, $S = 3.41$ | $R[F > 3\sigma(F)] = 0.062$, $wR(F) = 0.118$, $S = 1.64$ | $R[F > 3\sigma(F)] = 0.041$, $wR(F) = 0.088$, $S = 1.16$ | $R[F > 3\sigma(F)] = 0.041$, $wR(F) = 0.100$, $S = 1.64$ |
| No. of reflections | 5507 | 5013 | 2301 | 2953 |
| No. of parameters | 340 | 444 | 212 | 229 |
| No. of restraints | 0 | 0 | 0 | 0 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H-atom parameters constrained | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.80, −0.36 | 0.76, −0.63 | 0.11, −0.10 | 0.21, −0.18 |
| Absolute structure | – | – | 955 Friedel pairs used in the refinement | – |
| Absolute structure parameter | – | – | 0.5 | – |

| | TUJPEV | (VABZIJ) | WADXOR | YAXPOE |
|---|---|---|---|---|
| Crystal data | | | | |
| Chemical formula | $C_{10}H_{12}N_3O_3S^+\cdot C_7H_3N_2O_7^-$ | $C_8H_{13}N_2O^+\cdot C_7H_3N_2O_7^-\cdot H_2O$ | $C_9H_{17}N_2^+\cdot C_7H_3N_2O_7^-$ | $C_{26}H_{29}N_2^+\cdot C_7H_3N_2O_7^-$ |
| $M_r$ | 481.41 | 398.33 | 380.35 | 596.63 |
| Crystal system, space group | Triclinic, $P\overline{1}$ | Triclinic, $P\overline{1}$ | Monoclinic, $P2_1/n$ | Monoclinic, $P2_1/c$ |
| Temperature (K) | 296 | 100 | 200 | 200 |
| *a*, *b*, *c* (Å) | 8.5551 (1), 10.5000 (2), 12.7576 (3) | 6.6691 (3), 11.3831 (4), 12.2900 (5) | 6.1537 (3), 19.1541 (14), 14.5527 (11) | 14.5648 (3), 12.9374 (3), 16.1619 (3) |
| $\alpha$, $\beta$, $\gamma$ (°) | 106.463 (1), 100.913 (1), 108.272 (1) | 89.727 (2), 76.771 (2), 76.930 (2) | 90, 98.343 (6), 90 | 90, 103.900 (1), 90 |
| *V* (Å$^3$) | 993.72 (3) | 883.62 (6) | 1697.2 (2) | 2956.22 (11) |
| *Z* | 2 | 2 | 4 | 4 |
| Radiation type | Mo *K*$\alpha$ | Mo *K*$\alpha$ | Mo *K*$\alpha$ | Mo *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.23 | 0.13 | 0.12 | 0.10 |
| Crystal size (mm) | 0.20 × 0.20 × 0.16 | 0.52 × 0.13 × 0.10 | 0.30 × 0.13 × 0.10 | 0.51 × 0.26 × 0.17 |
| Data collection | | | | |
| Diffractometer | Bruker Kappa APEXII CCD | Bruker *SMART* APEXII CCD area-detector | Oxford Diffraction Gemini-S CCD-detector | Bruker APEXII CCD |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2004) | Multi-scan (*SADABS*; Bruker, 2009) | Multi-scan (*CrysAlis PRO*; Agilent, 2014) | Multi-scan (*SADABS*; Bruker, 2008) |
| $T_{min}$, $T_{max}$ | 0.955, 0.964 | 0.937, 0.987 | 0.920, 0.990 | 0.932, 1.000 |
| No. of measured, independent and observed $[I > 3\sigma(I)]$ reflections | 24261, 6717, 4398 | 17014, 4061, 3042 | 7800, 3339, 1976 | 29552, 7344, 5724 |
| $R_{int}$ | 0.030 | 0.030 | 0.034 | 0.015 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.758 | 0.650 | 0.617 | 0.667 |
| Refinement | | | | |
| *R* factors and goodness of fit | $R[F > 3\sigma(F)] = 0.044$, $wR(F) = 0.104$, $S = 1.95$ | $R[F > 3\sigma(F)] = 0.038$, $wR(F) = 0.086$, $S = 1.77$ | $R[F^2 > 2\sigma(F^2)] = 0.046$, $wR(F^2) = 0.095$, $S = 1.33$ | $R[F > 3\sigma(F)] = 0.054$, $wR(F) = 0.190$, $S = 1.80$ |
| No. of reflections | 6717 | 4061 | 3339 | 7344 |
| No. of parameters | 301 | 258 | 268 | 399 |
| No. of restraints | 0 | 0 | 2 | 0 |
| 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 | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.31, −0.35 | 0.46, −0.23 | 0.36, −0.24 | 0.63, −0.28 |
| Absolute structure | – | – | – | – |
| Absolute structure parameter | – | – | – | – |

and recalculated. Tables 1 and 2 contain an overview of those structures, which were successfully redetermined. In the following, these structures are referred to by their CSD refcodes; for the pertinent chemical names, see Table 2.

Notably, JEVNAA turns out not to be a substituted benzoate but a phenolate. NUQVEB though reported as a substituted benzoate turns out to be present in a disordered benzoate and a phenolate form. QIQJAD though reported as a disordered benzoate and a phenolate turns out to be a substituted benzoate. SAFGUD was reported as a substituted benzoate but turns out to be a phenolate. WADXOR was reported as a substituted benzoate that is disordered over two positions but it turns out to be present both in a dominant benzoate as well as in a minor phenolate form. Finally,

**Table 2**
Overview of the redetermined structures.

| REFCODE | Chemical name original/corrected if necessary |
|---|---|
| DUJZAK[a] | Bis(quinolin-8-ol)silver(I) 2-hydroxy-3,5-dinitrobenzoate |
| JEVNAA[b] | Tetrakis(1*H*-imidazole-$N^3$)zinc(II) bis(2-hydroxy-3,5-dinitrobenzoate / tetrakis(1*H*-imidazole-$N^3$)zinc(II) bis(2-carboxy-4,6-dinitrophenolate) |
| LUDFUL[c] | 1-Aza-8-azoniabicyclo[5.4.0]undec-7-ene 2-hydroxy-3,5-dinitrobenzoate / phenazine 2-hydroxy-3,5-dinitrobenzoic acid |
| NUQVEB[d] | 2-Amino-5-methylpyridinium 2-hydroxy-3,5-dinitrobenzoate) / 2-amino-5-methylpyridinium 2-hydroxy-3,5-dinitrobenzoate) (0.38) / 2-amino-5-methylpyridinium 2-carboxy-4,6-dinitrophenolate (0.62) |
| QIQJAD[e] | 3,5-Diamino-6-(2,3-dichlorophenyl)-1,2,4-triazin-2-ium 3,5-dinitro-2-hydroxybenzoate *N*,*N*-dimethylformamide solvate / 3,5-dinitro-2-hydroxybenzoate (0.55) 2-carboxy-4,6-dinitrophenolate (0.45) *N*,*N*-dimethylformamide monosolvate / 3,5-diamino-6-(2,3-dichlorophenyl)-1,2,4-triazin-2-ium 3,5-dinitro-2-hydroxybenzoate *N*,*N*-dimethylformamide monosolvate |
| SAFGUD[f] | Bis(1,10-phenanthroline-5,6-dione-2*N*,*N*′)silver(I) 2-hydroxy-3,5-dinitrobenzoate / bis(1,10-phenanthroline-5,6-dione-2*N*,*N*′)silver(I) 2-carboxy-4,6-dinitrophenolate |
| SEDKET[g] | 3,5-Dimethylpyrazolium 2-carboxy-4,6-dinitrophenolate / 3,5-dimethylpyrazolium 2-hydroxy-3,5-dinitrobenzoate |
| TIYZIM[h] | 3-(1*H*-Imidazol-1-yl)propanaminium 2-carboxy-4,6-dinitrophenolate |
| TUJPEV[i] | 4-[(5-methylisoxazol-3-yl)aminosulfonyl]anilinium 3,5-dinitrosalicylate |
| VABZIJ[j] | 2-Isopropyl-6-methyl-4-oxo-3,4-dihydropyrimidin-1-ium 2-carboxy-4,6-dinitrophenolatemonohydrate |
| WADXOR[k] | 1-Aza-8-azoniabicyclo[5.4.0]undec-7-ene 2-hydroxy-3,5-dinitrobenzoate / 2,3,4,6,7,8,9,10-octahydropyrimido[1,2-*a*] azepin-1-ium 2-hydroxy-3,5-dinitrobenzoate (0.73) / 2,3,4,6,7,8,9,10-octahydropyrimido[1,2-*a*]azepin-1-ium 2-carboxy-4,6–2-carboxy-4,6-dinitrophenolate (0.37) |
| YAXPOE[l] | 4-(Diphenylmethyl)-1-(3-phenylprop-2-en-1-yl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate |

Notes: (*a*) Zhang & Jian (2009); (*b*) Huang *et al.* (2007); (*c*) Senthil Kumar *et al.* (2002); (*d*) Hemamalini & Fun (2010*a*); (*e*) Sridhar *et al.* (2013); (*f*) Wang *et al.* (2012); (*g*) Wei *et al.* (2012); (*h*) Yamuna *et al.* (2014); (*i*) Malathy *et al.* (2015); (*j*) Hemamalini & Fun (2010*b*); (*k*) Smith & Lynch (2016); (*l*) Dayananda *et al.* (2012).

SEDKET was originally determined as a substituted phenolate but it turns out to be a benzoate.

Some of the retrieved structures were difficult or impossible to recalculate with sufficient accuracy: HILPOI (trimethoprimium 3,5-dinitrosalicylate; Subashini *et al.*, 2007) because of an abnormally low proportion of observed reflections (moreover the *bridging hydrogen* H6*a* is situated out of the plane between the carboxylate and hydroxy oxygen atoms, which seems to indicate an error) and VUZNEK (3,4-diaminopyridinium 2-carboxy-4,6-dinitrophenolate; Hemamalini & Fun, 2010*b*) because of the disorder present in the structure.

## 2. Refinement of the title structures

For each structure, two methods have been applied for the refinement of the hydrogen atoms involved in hydrogen bonding. In *Method 1*, the positions of the *bridging hydrogens* as well as those of the hydroxy, primary and secondary amine and ammonium hydrogen atoms were fixed after their localization in the difference electron-density maps while their displacement parameters were refined. In *Method 2*, the positional parameters of the latter hydrogen atoms were refined while their displacement parameters were constrained in the usual manner: $U_{iso}(H) = 1.2U_{eq}(N_{amine})$ or $U_{iso}(H) = 1.5U_{eq}(O_{hydroxy})$ or $U_{iso}(H) = 1.5U_{eq}(N_{ammonium})$.

The appropriate sections of the difference electron-density maps of the title structures (see supplementary Fig. S1) show regions with the hydroxy, amine and ammonium hydrogen atoms. These sections comprise the maps that were obtained after the refinement of the models without the pertinent hydrogen atoms as well as the maps that were calculated by either refinement method. It can be seen from the supplementary Fig. S1 that one of the reasons that hinders the correct localization of the hydrogen atoms involved in the hydrogen bonds is an apparent non-spherical electron density of the donor and acceptor atoms. Thus, hydrogen-atom localization by X-ray diffraction is hindered not only by its weak scattering power, but also by the polarization of its electron density resulting from the proximity of the acceptor and by the asphericity of the electron density of the donor and acceptor atoms. Therefore, refinement *Method 1* was given preference. The hydrogen bonds in the title structures are listed in Table 3, which shows that there might be quite a large difference between the results with the fixed and the refined positional parameters of such hydrogen atoms. In the following, a detailed description of the refinement of the recalculated structures is given:

**DUJZAK** (Zhang & Jian, 2009): C—$H_{aryl}$ were constrained to be equal to 0.93 Å while $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$. The position of the *bridging hydrogen* H3*b* as well as those of the hydroxy hydrogen atoms H1*aa* and H2*aa* were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined.

**JEVNAA** (Huang *et al.*, 2007): C—$H_{aryl}$ were constrained to be equal to 0.93 Å while $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$. The position of the *bridging hydrogen* H1*a* as well as those of the secondary amine hydrogen atoms H2*a* and H4*a* were located in the difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined.

**LUDFUL** (Senthil Kumar *et al.*, 2002): C—$H_{aryl}$ were constrained to be equal to 0.93 Å while $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$. The position of the *bridging hydrogen* H3*a* as well as that of the hydroxy hydrogen atom H1*a* were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined.

**NUQVEB** (Hemamalini & Fun, 2010*a*): The subroutine *TwinRotMax* of *PLATON* (Spek, 2009) indicated non-merohedral twinning: $h2 = -h1$; $k2 = -k1$; $l2 = -0.488\,h1 - 0.153k1 + l1$. The refinement was carried out on the non-overlapped reflections only. The refined value of the second domain fraction converged to the value −0.0006 (4). Therefore the value of the second domain fraction was set to 0 and was not refined further. C—$H_{aryl}$ and C—$H_{methyl}$ were constrained to be equal to 0.95 and 0.98 Å, respectively. $U_{iso}(H_{aryl})$ =

**Table 3**
Hydrogen bonds (Å, °) in the redetermined structures.

The upper entries for each hydrogen bond refer to refinement *Method 1*: fixed hydrogen-atom positions, which were obtained from the difference electron-density maps, and refined displacement parameters. The lower entries refer to refinement *Method 2*: refined hydrogen-atom positions and constrained displacement parameters.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| DUJZAK | | | | |
| O1—H1*aa*···O8 | 0.759 (2) | 1.859 (2) | 2.606 (3) | 167.96 (14) |
| | 0.97 (4) | 1.64 (4) | 2.603 (3) | 175 (3) |
| O2—H2*aa*···O9 | 0.922 (2) | 1.727 (2) | 2.631 (3) | 166.48 (15) |
| | 0.75 (4) | 1.90 (4) | 2.636 (3) | 165 (4) |
| O3—H3*b*···O9 | 1.040 (2) | 1.495 (2) | 2.481 (3) | 155.88 (12) |
| | 1.11 (4) | 1.41 (4) | 2.480 (3) | 160 (3) |
| JEVNAA | | | | |
| O2—H1*a*···O1 | 1.039 (2) | 1.496 (2) | 2.498 (2) | 160.4 (1) |
| | 0.89 (2) | 1.65 (3) | 2.503 (2) | 160 (2) |
| N2—H2*a*···O3 | 0.967 (2) | 1.890 (2) | 2.838 (3) | 165.9 (1) |
| | 0.84 (2) | 2.02 (2) | 2.845 (3) | 169 (2) |
| N4—H4*a*···O1$^{i}$ | 0.943 (2) | 1.924 (1) | 2.784 (2) | 150.6 (1) |
| | 0.86 (2) | 1.95 (2) | 2.792 (2) | 165 (2) |
| LUDFUL | | | | |
| O3—H3*a*···O2 | 1.059 (1) | 1.530 (1) | 2.513 (2) | 151.7 (1) |
| | 1.06 (2) | 1.51 (2) | 2.516 (2) | 156 (2) |
| O1—H1*a*···N3 | 1.163 (1) | 1.416 (1) | 2.552 (2) | 163.2 (1) |
| | 1.14 (2) | 1.44 (2) | 2.552 (2) | 166 (2) |
| NUQVEB | | | | |
| O7—H1*o*7···O1 | 0.919 (1) | 1.531 (1) | 2.4202 (12) | 161.55 (6) |
| | 1.14 (2) | 1.31 (2) | 2.4178 (12) | 163 (2) |
| O1—H1*o*1···O7 | 0.931 (1) | 1.513 (1) | 2.4202 (12) | 163.52 (6) |
| | 1.31 (2) | 1.14 (2) | 2.4178 (12) | 163 (2) |
| N2—H2*a*···O7$^{ii}$ | 0.892 (1) | 2.079 (1) | 2.9655 (14) | 172.84 (6) |
| | 0.87 (1) | 2.095 (14) | 2.9674 (14) | 176.5 (12) |
| N2—H2*b*···O1$^{iii}$ | 0.846 (1) | 2.165 (1) | 2.8526 (14) | 138.40 (6) |
| | 0.88 (2) | 2.146 (14) | 2.852 (1) | 137.3 (11) |
| N2—H2*b*···O2$^{iii}$ | 0.846 (1) | 2.413 (1) | 3.1741 (14) | 150.02 (6) |
| | 0.88 (2) | 2.384 (14) | 3.1736 (15) | 150.3 (11) |
| N1—H1···O6$^{ii}$ | 0.898 (1) | 1.783 (1) | 2.6781 (13) | 174.83 (6) |
| | 0.90 (1) | 1.784 (14) | 2.6773 (14) | 173.3 (13) |
| QIQJAD | | | | |
| N3—H3*n*···O2 | 0.862 (2) | 1.994 (2) | 2.854 (2) | 174.8 (1) |
| | 0.81 (3) | 2.05 (3) | 2.854 (3) | 175 (3) |
| N3—H4*n*···O8$^{iv}$ | 0.863 (2) | 2.059 (1) | 2.921 (2) | 176.9 (1) |
| | 0.85 (2) | 2.07 (2) | 2.920 (2) | 173 (3) |
| N2—H2*n*···O1 | 0.897 (2) | 1.831 (2) | 2.728 (2) | 177.5 (1) |
| | 0.81 (3) | 1.93 (3) | 2.731 (2) | 171 (2) |
| N5—H5*n*···N4$^{v}$ | 0.866 (1) | 2.141 (1) | 2.9992 (19) | 171.1 (1) |
| | 0.84 (2) | 2.17 (2) | 2.999 (2) | 171 (2) |
| N5—H6*n*···O8$^{vi}$ | 0.863 (2) | 2.041 (2) | 2.760 (2) | 140.2 (1) |
| | 0.78 (2) | 2.12 (3) | 2.764 (2) | 141 (2) |
| O3—H3*o*···O1 | 0.926 (1) | 1.562 (1) | 2.4572 (18) | 161.3 (1) |
| | 0.99 (3) | 1.49 (3) | 2.4569 (19) | 164 (3) |
| SAFGUD | | | | |
| O8—H7···O7 | 1.155 (4) | 1.346 (4) | 2.462 (6) | 159.6 (3) |
| | 1.05 (7) | 1.57 (7) | 2.452 (7) | 138 (6) |
| SEDKET | | | | |
| O1—H2*a*···O2 | 1.22 (5) | 1.34 (5) | 2.476 (3) | 149 (5) |
| | 1.27 (3) | 1.29 (3) | 2.477 (3) | 151 (3) |
| O2—H2*a*···O1 | 1.34 (5) | 1.22 (5) | 2.476 (3) | 149 (5) |
| | 1.29 (3) | 1.27 (3) | 2.477 (3) | 151 (3) |
| N1—H1···O1$^{vii}$ | 1.11 (5) | 1.92 (5) | 2.799 (4) | 133 (3) |
| | 0.99 (4) | 2.00 (3) | 2.804 (4) | 137 (3) |
| N1—H1···O7$^{vii}$ | 1.11 (5) | 1.94 (5) | 2.850 (4) | 137 (3) |
| | 0.99 (4) | 2.03 (3) | 2.855 (4) | 140 (3) |
| N2—H2···O3 | 0.96 (3) | 1.77 (3) | 2.685 (4) | 158 (3) |
| | 0.99 (3) | 1.75 (3) | 2.684 (4) | 157 (3) |

**Table 3 (continued)**

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| TIYZIM | | | | |
| O2*b*—H2*b*···O1*b* | 0.982 (1) | 1.516 (1) | 2.4473 (16) | 156.3 (1) |
| | 1.02 (2) | 1.48 (2) | 2.4476 (16) | 156 (2) |
| N3*a*—H3*aa*···N1*aa*$^{viii}$ | 0.904 (1) | 1.932 (1) | 2.797 (2) | 159.6 (1) |
| | 0.91 | 1.92 | 2.797 (2) | 162 |
| N3*a*—H3*ab*···O2*b*$^{ix}$ | 0.901 (1) | 2.565 (1) | 3.1297 (17) | 121.4 (1) |
| | 0.91 | 2.58 | 3.1298 (17) | 120 |
| N3*a*—H3*ab*···O2*b*$^{ix}$ | 0.901 (1) | 2.565 (1) | 3.1297 (17) | 121.4 (1) |
| | 0.91 | 2.58 | 3.1297 (18) | 120 |
| N3*a*—H3*ab*···O3*b*$^{ix}$ | 0.901 (1) | 2.072 (1) | 2.9537 (17) | 165.8 (1) |
| | 0.91 | 2.06 | 2.9542 (17) | 165 |
| N3*a*—H3*ac*···O1*b*$^{x}$ | 0.893 (1) | 2.061 (1) | 2.815 (2) | 141.5 (1) |
| | 0.91 | 2.03 | 2.815 (2) | 144 |
| N3*a*—H3*ac*···O7*b*$^{x}$ | 0.893 (1) | 2.484 (1) | 2.9712 (19) | 114.7 (1) |
| | 0.91 | 2.46 | 2.9706 (19) | 116 |
| TUJPEV | | | | |
| O6—H6*a*···O5 | 1.184 (1) | 1.295 (1) | 2.4268 (16) | 156.58 (6) |
| | 1.24 (2) | 1.21 (2) | 2.4280 (17) | 165.3 (14) |
| N1—H1*a*···O6$^{xi}$ | 1.002 (1) | 2.068 (1) | 3.0655 (17) | 173.55 (7) |
| | 0.89 | 2.24 | 3.0694 (17) | 155 |
| N1—H1*b*···N3$^{v}$ | 0.793 (1) | 2.292 (1) | 3.0393 (15) | 157.3 (1) |
| | 0.89 | 2.20 | 3.0382 (15) | 157 |
| N1—H1*c*···O4$^{v}$ | 0.832 (2) | 1.831 (1) | 2.663 (2) | 177.1 (1) |
| | 0.89 | 1.77 | 2.660 (2) | 175 |
| N2—H2*a*···O5 | 0.970 (1) | 1.844 (1) | 2.7852 (15) | 162.64 (9) |
| | 0.827 (17) | 1.986 (16) | 2.7900 (16) | 164.0 (18) |
| VABZIJ | | | | |
| N3—H1*n*3···O6$^{x}$ | 0.973 (1) | 1.754 (1) | 2.7182 (14) | 170.48 (8) |
| | 0.91 (1) | 1.823 (14) | 2.7214 (14) | 170.8 (15) |
| N4—H1*n*4···O1*w* | 0.909 (1) | 1.840 (1) | 2.7348 (15) | 167.76 (8) |
| | 0.91 (2) | 1.833 (15) | 2.7323 (16) | 172.0 (15) |
| O1*w*—H2*w*1···O1$^{xii}$ | 0.917 (1) | 1.890 (1) | 2.7886 (14) | 166.21 (6) |
| | 0.82 (2) | 1.995 (19) | 2.7906 (15) | 162.7 (16) |
| O1*w*—H1*w*1—O3$^{iii}$ | 0.915 (1) | 2.040 (1) | 2.9352 (14) | 165.84 (7) |
| | 0.89 (2) | 2.064 (18) | 2.9357 (15) | 168.0 (17) |
| O7—H7···O1 | 1.019 (1) | 1.433 (1) | 2.4340 (13) | 165.94 (7) |
| | 0.96 (2) | 1.505 (16) | 2.4358 (13) | 162.0 (16) |
| WADXOR | | | | |
| N8*a*—H8*a*···O11*b* | 0.960 (2) | 1.933 (2) | 2.864 (2) | 162.83 (11) |
| | 0.91 (2) | 1.96 (2) | 2.869 (2) | 174.1 (17) |
| O11*b*—H21*b*···O21*b* | 1.145 (2) | 1.303 (6) | 2.433 (6) | 167.3 (3) |
| | 1.07 (9) | 1.48 (9) | 2.430 (6) | 145 (7) |
| O2*b*—H2*b*···O12*b* | 1.103 (2) | 1.385 (2) | 2.471 (2) | 166.81 (13) |
| | 0.91 (3) | 1.61 (3) | 2.475 (3) | 159 (3) |
| YAXPOE | | | | |
| N1—H71···O1$^{iv}$ | 0.945 (1) | 1.954 (1) | 2.813 (2) | 150.01 (8) |
| | 0.90 (2) | 1.98 (2) | 2.812 (2) | 154.0 (19) |
| N1—H71···O2$^{iv}$ | 0.945 (1) | 2.302 (2) | 3.032 (2) | 133.62 (8) |
| | 0.90 (2) | 2.36 (2) | 3.034 (2) | 131.8 (17) |
| O7—H7···O1 | 0.924 (2) | 1.668 (1) | 2.505 (2) | 148.96 (9) |
| | 0.92 (3) | 1.71 (3) | 2.504 (2) | 142 (2) |

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y, -z$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+1, -y+2, -z+1$; (vi) $x-1, y+1, z$; (vii) $-x+1, y+\frac{1}{2}, -z+1$; (viii) $-x, -y, -z$; (ix) $x+1, y, z$; (x) $-x, -y+1, -z+1$; (xi) $-x, -y+2, -z+1$; (xii) $x, y+1, z$.

$1.2U_{eq}(C_{aryl})$ and $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$. The positions of the disordered *bridging hydrogens* H1*o*1 and H1*o*7 as well as those of the primary (H2*a*, H2*b*) and the secondary amine hydrogen atoms (H1*a*) were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined; in the case of the *bridging hydrogens* H1*o*1 and

H1*o*7, their isotropic displacement parameters were refined to be equal while their occupational parameters were refined under the condition that their sum was equal to 1.

**QIQJAD** (Sridhar *et al.*, 2013): The subroutine *TwinRotMax* of *PLATON* (Spek, 2009) indicated non-merohedral twinning: $h2 = -1.018h1 + 0.054k1$; $k2 = -0.673h1 + 1.018k1$; $l2 = -0.039h1 + 0.116k1 - l1$. The refined value of the second domain fraction converged to the value 0.028 (13). Therefore the value of the second domain fraction was set to 0 and was not refined further. C—H$sp^2$ and C—$H_{methyl}$ were constrained to equal to 0.93 and 0.96 Å, respectively. $U_{iso}$(H$sp^2$) = 1.2$U_{eq}$(C$sp^2$) and $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$. The positions of the *bridging hydrogen* H3*o* and those of the primary (H3*n*, H4*n*, H5*n*, H6*n*) as well as of the secondary (H2*n*) amine hydrogen atoms were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined.

**SAFGUD** (Wang *et al.*, 2012)**:** C—$H_{aryl}$ were constrained to be equal to 0.93 Å while $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$. The *bridging hydrogen* H7 was located in a difference electron-density map and its position was fixed while its isotropic displacement parameter $U_{iso}$(H7) was refined.

**SEDKET (**Wei *et al.*, 2012): The non-centrosymmetric structure is composed of the light atoms only (the heaviest atom is O) and the data collection was carried out with Mo *K*α radiation. The article by Wei *et al.* (2012) does not indicate whether the Friedel pairs were merged and nor does it contain the value of the Flack parameter. The Flack parameter was set to 0.5 without being refined in the present model. C—$H_{aryl}$ and C—$H_{methyl}$ were constrained to be equal to 0.93 and 0.96 Å, respectively. $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$ and $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$. The position of the *bridging hydrogen* H2*a* as well as those of the secondary amine hydrogen atoms H1 and H2 were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined.

**TIYZIM** (Yamuna *et al.* (2014): C—$H_{aryl}$ and C—$H_{methylene}$ were constrained to be equal to 0.95 and 0.99 Å, respectively. $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$ and $U_{iso}(H_{methylene}) = 1.5U_{eq}(C_{methylene})$. The position of the *bridging hydrogen* H2*b* as well as those of the ammonium hydrogen atoms (H3*aa*, H3*ab*, H3*ac*) were found in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined; in the case of the ammonium hydrogen atoms (H3*ab*, H3*ac*,), their displacement parameters were constrained to be equal to that of H3*aa*.

**TUJPEV** (Malathy *et al.*, 2015): C—$H_{aryl}$ were constrained to be equal to 0.93 Å while $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$. C—$H_{methyl}$ were constrained to be equal to 0.96 Å while $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$. The position of the *bridging hydrogen* H6*a* as well as those of the secondary amine group H2*a* and of the ammonium hydrogen atoms H1*a*, H1*b* and H1*c* were found in a difference-electron map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined; in the case of the ammonium hydrogen atoms (H1*b*, H1*c*), their displacement parameters were constrained to be equal to that of H1*a*.

**VABZIJ** (Hemamalini & Fun, 2010*c*): C—$H_{aryl}$, C—$H_{methyl}$, C—$H_{methine}$ were constrained to be equal to 0.93, 0.96 and 0.98 Å, respectively. $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$, $U_{iso}(H_{methine}) = 1.2U_{eq}(C_{methine})$, $U_{iso}(H_{methyl}) = 1.5U_{eq}(C_{methyl})$. The position of the *bridging hydrogen* H7 as well as those of the secondary amine hydrogen atom H1*n*4 and of the water hydrogen atoms H1*w*1 and H1*w*2 were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their displacement parameters were refined.

**WADXOR** (Smith & Lynch, 2016): The non-centrosymmetric structure is composed of light atoms only (the heaviest atoms present in the structure are oxygens) and the data collection was carried out with Mo *K*α radiation. The original article reported the refined Flack parameter to be equal to −0.1 (13); however, the refinement using *JANA*2006 (Petříček *et al.*, 2014) did not converge and therefore the Flack parameter was set to 0.5 without being refined. C—$H_{aryl}$ and C—$H_{methylene}$ were constrained to be equal to 0.95 and 0.99 Å, respectively, except for the distances between the methylene atom C11 and the attached hydrogen atoms H12*a* and H13*a*, which were restrained to 0.99 (1) Å (Müller, 2009). [The reason for the different treatment of the latter methylene group was its vicinity to the disordered methylene groups centered on C10 and C12*a*.] $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$ and $U_{iso}(H_{methylene}) = 1.2U_{eq}(C_{methylene})$. There were two types of occupational disorder present in the structure. The first one was related to the fragments with the methylene carbon atoms C9*a*, C10*a* and the attached respective pairs of hydrogen atoms H91*a*, H92*a* and H10*a*, H11*a*, as well as to C13*a* and C12*a* with the attached respective pairs of hydrogen atoms H16*a*, H17*a* and H14*a*, H15*a*. The occupation parameter of C13 was refined while those of the related atoms were either set equal to that of C13 (*i.e.* C12*a* and attached hydrogen atoms) or its complement to 1 (C9*a* and C10*a* and attached hydrogen atoms). The displacement parameters of the disordered pairs of atoms C9*a* and C13*a* as well as C10*a* and C12*a* were set to be equal, *i.e.* that of C13*a* equalled that of C9*a* while that of C10*a* equalled that of C12*a*. The second type of occupational disorder referred to the fragments C2*b*—H61*b*, C2*b*–O2*b*—H2*b* and C6*b*—H6*b*, C6*b*—O21*b*—H21*b*. This means that the occupation parameters of H61*b*, H21*b* were set equal to the refined occupational parameter of O21*b* while being complements to 1 for H6*b*, O2*b*, H2*b*. The positions of the *bridging hydrogens* H2*b* and H21*b* as well as that of the primary amine hydrogen atom H8*a* were located in a difference electron-density map. Their positional parameters were fixed during the refinement while their isotropic displacement parameters were refined; in the case of *bridging hydrogens* H2*b* and H21*b*, their isotropic displacement parameters were constrained to be equal.

**YAXPOE** (Dayananda *et al.*, 2012): C—$H_{aryl}$ and C—$H_{methylene}$ were constrained to equal to 0.95 and 0.99 Å, respectively. $U_{iso}(H_{aryl}) = 1.2U_{eq}(C_{aryl})$ and $U_{iso}(H_{methylene}) = 1.5U_{eq}(C_{methylene})$. The *bridging hydrogen* H7 was located in a difference electron-density map. Its positional parameters

were fixed while $U_{iso}$(H7*a*) was refined. A high instability factor $\Delta$ in the weighting scheme (0.0064) was applied in order to avoid a large number of reflections with $(I_{obs} - I_{calc})/\sigma(w) > 10$ where $\sigma(w) = [\sigma^2(I) + \Delta I^2]^{-1/2}$. [This condition generates A alerts for $\Delta = 0.0004$, which has been used in other refinements of the title structure, when running *checkCIF* (Spek, 2009).] The residual electron-density map contains peaks which are difficult to interpret (see supplementary Fig. S1).

## 3. Discussion of the interdependence of bond lengths and angles

For this discussion, the definition of the various bonds and angles in the moieties of **I**–**IV** (shown in the scheme), are illustrated in Figs. 1*a* and 1*b*, respectively. As already pointed out, the dependence *D*2 on *D*4 and *D*1 on *D*3 (Fig. 2) has shown that a large number of structures are biased by incorrectly applied constraints or restraints on the *bridging hydrogen*. However, a dubious or incorrect localization of the

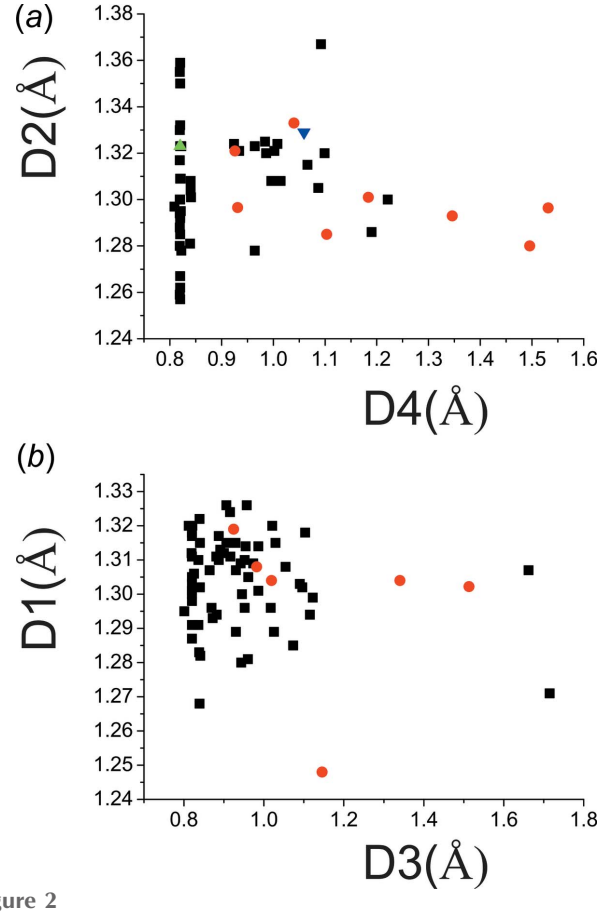



**Figure 2**
The dependence of bond distances: (*a*) *D*2 on *D*4 for structures that were originally determined as 2-hydroxy-3,5-dinitrobenzoate (**II**), or as containing 2-hydroxy-3,5-dinitrobenzoic acid (**I**); (*b*) *D*1 on *D*3 for the structures that were determined as 2-carboxy-4,6-dinitrophenolate (**III**). Colour code for symbols: black squares are the data retrieved from the CSD; red circles are the corrected title structures; green and blue triangles are the original and the corrected structure of LUDFUL, which contains a molecule of 2-hydroxy-3,5-dinitrobenzoic acid (**I**).

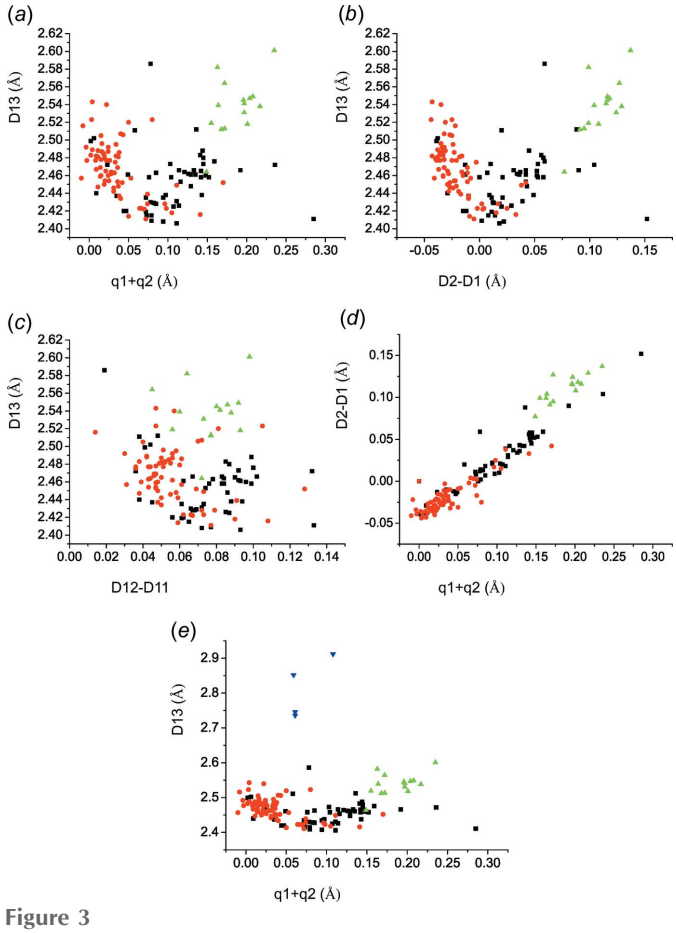



**Figure 3**
The dependence of distances: (*a*) *D*13 on (*q*1 + *q*2); (*b*) *D*13 on *D*2 − *D*1; (*c*) *D*13 on *D*12—*D*11; (*d*) *D*2 − *D*1 on (*q*1 + *q*2); (*e*) *D*13 on (*q*1 + *q*2), also for the structures with 3,5-dinitro-2-oxidobenzoate (**IV**), which are shown as blue triangles. Colour code for symbols: green triangles refer to the structures with 2-hydroxy-3,5-dinitrobenzoic acid (**I**), black squares are the structures with 2-hydroxy-3,5-dinitrobenzoate (**II**), and red circles are the structures with 2-carboxy-4,6-dinitrophenolates (**III**).

*bridging hydrogen* or the acid hydrogen is believed to affect the positions of the non-hydrogen atoms only minutely, and therefore even the biased structures can be considered further. The parameters $q1 = D2 - D1$ and $q2 = D12 - D11$ express the electron delocalization within the fragment *D*1–*D*12–*D*11–*D*2. The introduction of the parameters *q*1 and *q*2 follows an analogous discussion of resonance-assisted hydrogen bonds in the enol forms of $\beta$-diketone fragments (Gilli *et al.*, 1989, 2009). Fig. 3*a* shows that the distance where the structures with 2-carboxy-4,6-dinitrophenolates (**III**; red circles) transform into 2-hydroxy-3,5-dinitrobenzoates (**II**; black squares) corresponds to the shortest distance $D13_{min} \simeq 2.41$ Å, which in turn corresponds to $(q1 + q2) \simeq 0.08$ Å. This implies that this is the region where the *bridging hydrogen* has the greatest tendency to be situated about the centre of the O···O intramolecular hydrogen bond or disordered about it. A very similar dependence is shown in Fig. 3*b*, where only distances *D*1 and *D*3 are compared. The observed dependence means that the elongation of one C—O bond takes place mostly at the cost of the shortening of the neighbouring C═O bond; in other words, the distance between these two O atoms,

D13 $\simeq [(D13_{min})^2 + (D2 - D1)^2]^{1/2}$ (Fig. 1). Table 4 lists the structures in which the title molecules are present in different forms. In the recalculated structure of SEDKET (Table 2) and *e.g.* the reported structures of KEZJIJ (Song *et al.*, 2007) and KEZJIJ01 (Smith *et al.*, 2007) that refer to the structure determination of 2-(pyridin-2-yl)pyridinium 2-carboxy-4,6-dinitrophenolate, the *bridging hydrogen* is attached to the O atom having the shorter C—O bond distance.

Fig. 3*a* and 3*b* also show that the *bridging hydrogen* cannot be situated near the centre of the intramolecular O···O hydrogen bond in structures with 2-hydroxy-3,5-dinitrobenzoic acid (**I**). Fig. 3*c* shows a similar dependence of *D*13 on (*D*12 − *D*11). It can be seen that the adjacent C—C conjugated bonds are less, but still sensitive to the bonding of the hydroxy hydrogen atom to one of the neighbouring C—O groups. These properties indicate that the O···H···O hydrogen bonding with the pertinent O···O distance *D*13 belongs to the category of resonance-assisted hydrogen bonds (Gilli *et al.*, 1989, 2009; Sobczyk *et al.*, 2005).

Fig. 3*d* compares both dependences shown in Figs. 3*a* and 3*b*. It can be seen that the dependence of (*D*2 − *D*1) on (*q*1 + *q*2) is fairly linear. The dependence seems to show the narrowest spread for the 2-hydroxy-3,5-dinitrobenzoates (**II**),

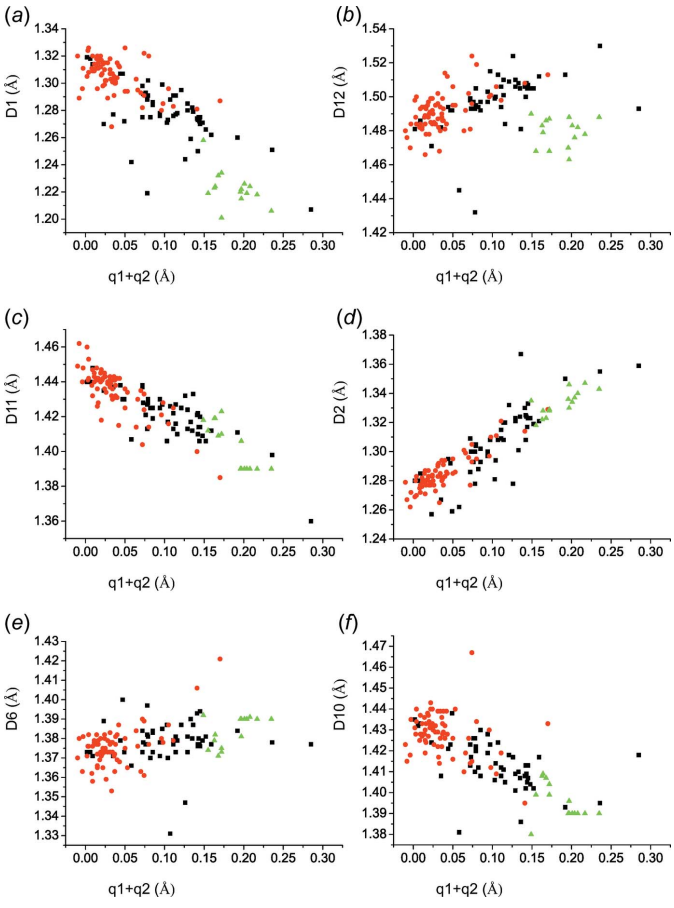



**Figure 4**
The dependence of bond distances: (*a*) *D*1 on (*q*1 + *q*2); (*b*) *D*12 on (*q*1 + *q*2); (*c*) *D*11 on (*q*1 + *q*2); (*d*) *D*2 on (*q*1 + *q*2); (*e*) *D*6 on (*q*1 + *q*2); (*f*) *D*10 on (*q*1 + *q*2). The colour code for the symbols is the same as in Fig. 3.

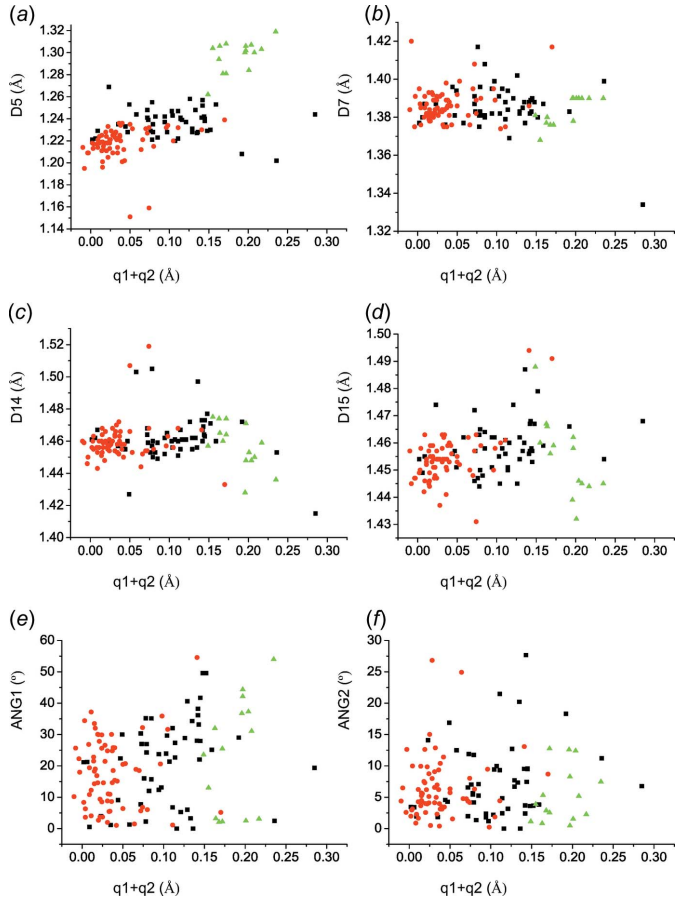



**Figure 5**
The dependence of bond distances: (*a*) *D*5 on (*q*1 + *q*2); (*b*) *D*7 on (*q*1 + *q*2); (*c*) *D*14 on (*q*1 + *q*2). The dependence of dihedral angles: (*e*) *ANG*1 on (*q*1 + *q*2); (*f*) *ANG*2 on (*q*1 + *q*2). [*ANG*1 and *ANG*2 are the dihedral angles of the nitro groups involving bonds *D*14 and *D*15, respectively, toward the ring plane.] The colour code of the symbols is the same as in Fig. 3.

which are represented by the black squares. Importantly, the line for each class of molecules intercepts the *D*2 − *D*1 axis at different values. The structures that contain 2-hydroxy-3,5-dinitrobenzoic acid (**I**) molecules (green triangles) are clearly separated from the rest of the structures although they show a similar trend. Figs. 3*a*–3*d* also show outliers that do not fit the overall trends and which are most probably the structures determined as 2-hydroxy-3,5-dinitrobenzoates (**II**) instead of 2-carboxy-4,6-dinitrophenolates (**III**) and *vice versa*. Fig. 3*e* shows the same as Fig. 3*a* except for the addition of a few known structures that contain a 3,5-dinitro-2-oxidobenzoate (**IV**), which are indicated by blue triangles. Their positions can be explained by the fact that the carboxylate groups are substantially inclined to the benzene ring in such compounds, which causes elongation of the distance between the carboxylate and oxo group, and these molecules will not be considered further.

The alternation of the inclinations (Fig. 4*a*–4*d*) of the dependences of *D*1, *D*12, *D*11, and *D*2 on (*q*1 + *q*2) are in agreement with the delocalization of the electron density in these bonds. The 2-hydroxy-3,5-dinitrobenzoic acid (**I**) mol-

ecules (green triangles) and the 2-hydroxy-3,5-dinitrobenzoates (**II;** black squares) are situated apart from the 2-carboxy-4,6-dinitrophenolates (**III;** red circles) in the given figures. The fact that *D*1 tends to be shortest in 2-hydroxy-3,5-dinitrobenzoic acid (**I**) molecules (Fig. 4*a*) can be explained by the elongation of bond *D*5 in the latter molecules because of the attachment of the hydrogen atom and the concomitant shortening of *D*1. The bond lengths *D*1 (Fig. 4*a*) are equal to 1.28–1.30 Å at $(q1 + q2) \simeq 0.08$ where the highest probability for the occurrence of a symmetric intramolecular O···H···O hydrogen bond takes place. The corresponding values of *D*12, *D*11, *D*2, *D*6 and *D*10 are 1.49 Å (Fig. 4*b*), 1.43 Å (Fig. 4*c*), 1.30 Å (Fig. 4*d*), 1.37–1.39 Å (Fig. 4*e*) and 1.41–1.43 Å (Fig. 4*f*).

Fig. 5*a* shows the dependence of *D*5 on $(q1 + q2)$. Comparing Fig. 5*a* to Fig. 4*a*, which shows the dependence of *D*1 on $(q1 + q2)$, an indirect proportionality of both dependences can be observed. The bond length *D*5 is equal to 1.22–1.24 Å for $(q1 + q2) \simeq 0.08$ Å. The dependence of *D*5 on $(q1 + q2)$ (Fig. 5*a*) is similar to that of bond *D*12 (Fig. 4*b*) in 2-hydroxy-3,5-dinitrobenzoates (**II**) and 2-carboxy-4,6-dinitrophenolates (**III**), but not in molecules of 2-hydroxo-3,5-dinitrobenzoic acid (**I**). It is interesting that 2-hydroxy-3,5-dinitrobenzoic acid (**I**) molecules are in line with other forms of the title molecules for the dependences in Fig. 5*c* and Fig. 4*d*. Bond *D*7 is rather distant from the carboxylic group (Fig. 5*b*) and the delocalization within the pyridine ring is no longer clear. The same holds for bonds *D*14 and *D*15 (Figs. 5*c* and 5*d*). Figs. 5*e* and 5*f* show the inclinations, *ANG*1 and *ANG*2, of the nitro groups involving bonds *D*14 and *D*15, respectively, toward the ring plane.

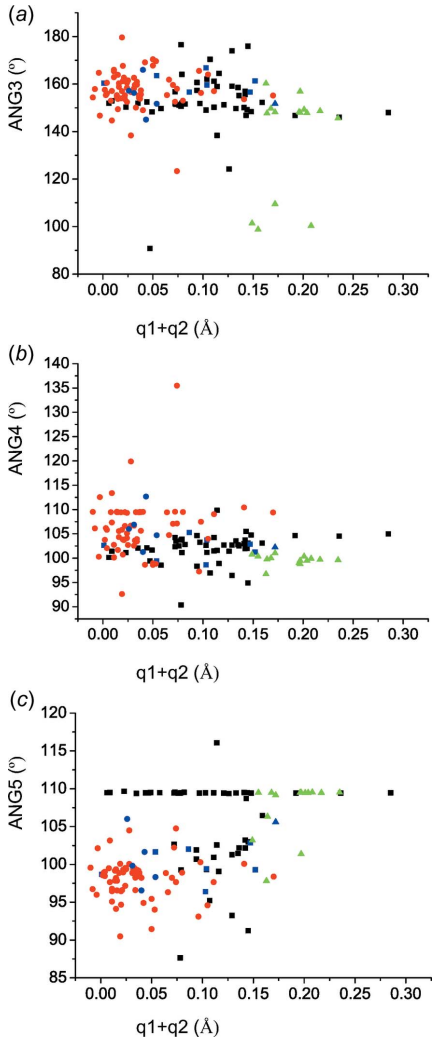



**Figure 6**
(*a*) Dependence of the O···H···O angle *ANG*3 on $(q1 + q2)$; (*b*) dependence of *ANG*4 on $(q1 + q2)$; (*c*) dependence of *ANG*5 on $(q1 + q2)$. Colour code for symbols: green triangles refer to the structures with 2-hydroxy-3,5-dinitrobenzoic acid (**I**), black squares are the structures with 2-hydroxy-3,5-dinitrobenzoate (**II**), and red circles are the structures with 2-carboxy-4,6-dinitrophenolate (**III**); blue triangles, squares and circles are the recalculated structures with 2-hydroxy-3,5-dinitrobenzoic acid (**I**), 2-hydroxy-3,5-dinitrobenzoate (**II**) and 2-carboxy-4,6-dinitrophenolate (**III**), respectively.

Figs. 6*a*–6*c* show dependences in which the localization of the *bridging hydrogen* takes place. It seems that the most obtuse angles of O···H···O (*ANG*3) occur for $(q1 + q2)$ in the range <0.06–0.10> Å, *i.e.* for the shortest distances of *D*13 (2.41 Å). It is questionable whether the position of a *bridging hydrogen* in the transition zone between 2-hydroxy-3,5-dinitrobenzoates (**II**) and 2-carboxy-4,6-dinitrophenolates (**III**) facilitates its positional disorder, which occurs *e.g.* in NUQVEB, because of the impossibility of angle *ANG*3 approaching 180°. The dependence of the angles *ANG*4 and *ANG*5 (Fig. 1*b*) shows once more the effect of incorrectly applied constraints, which are manifested by values close to 109.54° (*cf.* Figs. 2*a* and 2*b*).

The previous discussion has shown the correlations of *D*1 and *D*5 on $(q1 + q2)$ (Figs. 4*a* and 5*a*, respectively), and the indirect dependence of *D*1 on *D*5. Therefore, the position of the *bridging hydrogen* is expected to be related to the environment of the molecules, *i.e.* to be dependent on $\Delta pK_a = pK_a(\text{base}) - pK_a(\text{acid})$. The value of $\Delta pK_a$ is correlated with the occurrence of a structure where the base and the acid components are not ionized, thus forming a co-crystal ($\Delta < 0$), or ionized forming a salt ($\Delta pK_a > 3$) (Childs *et al.*, 2007). It is difficult to predict the form in which the acid and the base are present for $0 < \Delta pK_a < 3$ (Childs *et al.*, 2007).

In Table 4, the structures are ordered according to ascending values of the $pK_a$ values of the bases, *i.e.* according to increasing basicity. The corresponding values of $\Delta pK_a$ are compared with $(q1 + q2)$ and *D*13. The $pK_a$ of 2-hydroxy-3,5-dinitrobenzoic acid (**I**; 3,5-dinitrosalicylic acid) is reported as 2.18 (Smith & Wermuth, 2014; Hemamalini & Fun, 2010*a*), although a value of 1.53 has been reported in the literature (https://www.chemicalbook.com/ProductMSDSDetailCB9172047_EN.htm). The weakest bases given at the top of Table 4 are not able to deprotonate the title molecule, which remains in the form of 2-hydroxy-3,5-dinitrobenzoic acid (**I**). On the other hand, the bases with the largest values of $pK_a$ (see the bottom of Table 3) are able to deprive the title molecule of the hydroxy and acid hydrogen atoms, so in such cases the resulting molecule would be in the form of 3,5-dinitro-2-oxidobenzoate (**IV**). The compounds with moderate basicities are able to deprotonate the acid hydrogen atom but not the *bridging hydrogen;* hence, the resulting forms are 2-hydroxy-

**Table 4**
Overview of selected structures with different forms of the molecules: 2-hydroxy-3,5-dinitrobenzoic acid (**I**); 2-hydroxy-3,5-dinitrobenzoate (**II**); 2-carboxy-4,6-dinitrophenolate (**III**); 3,5-dinitro-2-oxidobenzoate (**IV**).

The structures are ordered by ascending p$K_a$ value of the base. The corresponding values of ($q1 + q2$), $D13$, $D1$, $D2$ and $D5$ (*cf.* Fig. 1) are also given.

| | Refcode | Base and its form present in the structure | p$K_a$ | $\Delta$p$K_a$ | Type | ($q1 + q2$) (Å) | $D13$ (Å) | $D1$ (Å) | $D2$ (Å) | $D5$ (Å) | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | GORXA*M*[a] | 1,4-dioxane | −3.9 | −6.08 | **I** | 0.204<br>0.196 | 2.547<br>2.545 | 1.219<br>1.220 | 1.337<br>1.336 | 1.307<br>1.300 | Two independent molecules |
| 2 | GORXEQ[a] | 1,4-dioxane | −3.9 | −6.08 | **I** | 0.235 | 2.601 | 1.206 | 1.343 | 1.319 | |
| 3 | GORXEQ01[a] | 1,4-dioxane | −3.9 | −6.08 | **I** | 0.197 | 2.531 | 1.222 | 1.346 | 1.302 | |
| 4 | AJEBOG[b] | 4-cyano-pyridinium | 1.92 | −0.26 | **III** | 0.003 | 2.523 | 1.324 | 1.28 | 1.213 | |
| 5 | ABULA*M*[c] | 2-amino-anilinium | <2 | <-0.18 | **III** | 0.011 | 2.447 | 1.309 | 1.282 | 1.219 | |
| 6 | PIDCAI[c] | 2-amino-anilinium | <2 | <-0.18 | **III** | 0.009 | 2.44 | 1.314 | 1.285 | 1.229 | Wrongly attached hydrogen due to C═O distances. Originally determined as type **II** but it should be **III**. |
| 7 | PERBA*R*[d] | 3-carbamoyl-pyridinium | 3.35 | 1.2 | **II** | 0.17 | 2.452 | 1.287 | 1.329 | 1.239 | Wrongly attached hydrogen due to C═O distances. Originally determined as type **II** but it is probably **III**. Disorder present in the structure. |
| 8 | GIFMUE[e] | 1-naphthyl-ammonium | 3.92 | 1.74 | **III** | 0.011 | 2.488 | 1.31 | 1.279 | 1.224 | |
| 9 | MIPROS[f] | 8-amino-quinolinium | 3.95 | 1.77 | **II** | 0.072 | 2.408 | 1.278 | 1.300 | 1.237 | The *bridging hydrogen* is situated about the centre. |
| 10 | ABUKUF[g] | 4-chloro-anilinium | 3.98 | 1.80 | **II** | 0.094 | 2.435 | 1.276 | 1.297 | 1.242 | |
| 11 | YIVHIW[h] | 4-iodo-anilinium | 4.18 | 1.63 | **II** | 0.129 | 2.461 | 1.285 | 1.321 | 1.228 | |
| 12 | GIFNUF[i] | 1,10-phenan-throlinium | 4.27 | 2.09 | **II** | 0.096 | 2.428 | 1.280 | 1.297 | 1.232 | Determined as the type **III** but it is probably **II** (Fig. 1). The chemical name was correct. |
| 13 | FOXHAD[j] | 2-(pyridin-2-yl)-pyridinium | 4.33 | 2.15 | **II** | 0.047 | 2.42 | 1.307 | 1.292 | 1.228 | 100 K; the reported hydrogen H3 is situated out of the plane formed by C···O bonds and is superficial. |
| 14 | KEZJIJ[j] | 2-(pyridin-2-yl)-pyridinium | 4.33 | 2.15 | **III** | 0.07 | 2.422 | 1.293 | 1.296 | 1.231 | C═O distances are about equal. The recalculation has shown that the *bridging hydrogen* is about the centre of the hydrogen bond, slightly closer to atom O2, which forms a shorter C═O bond. |
| 15 | KEZJIJ01[j] | 2-(pyridin-2-yl)-pyridinium | 4.33 | 2.15 | **III** | 0.066 | 2.423 | 1.295 | 1.299 | 1.221 | C═O distances are about equal, the hydrogen is attached to the O atom forming a shorter C═O bond. |
| 16 | FICXIZ[k] | cytosinium | 4.60 | 2.42 | **II** | 0.098 | 2.423 | 1.285 | 1.310 | 1.234 | The type according to the C═O distances should be **II**; the *bridging hydrogen* was wrongly attached. |
| 17 | ABUJUE[l] | anilinium | 4.60 | 2.42 | **II** | 0.129 | 2.448 | 1.280 | 1.323 | 1.231 | |
| 18 | ABUKO*Z*[m] | 4-fluoro-anilinium | 4.65 | 2.47 | **II** | 0.142 | 2.465 | 1.273 | 1.325 | 1.252 | |
| 19 | GIFMOY[n] | quinolinium | 4.85 | 2.67 | **III** | 0.05 | 2.414 | 1.294 | 1.285 | 1.235 | The title molecule has similarly long C═O distances. |
| 20 | ZAJHAT[o] | 2-ammino-benzoic acid | 4.96 | 2.78 | **II** | 0.135 | 2.461 | 1.282 | 1.324 | 1.227 | |
| 21 | AJEBIA[p] | pyridinium | 5.23 | 3.05 | **I** and **II** | 0.142<br>0.163 | 2.458<br>2.582 | 1.250 | 1.308 | 1.257 | Two independent molecules |
| 22 | EGABOF[q] | 2-methyl-quinolinium | 5.71 | 3.53 | **II** | 0.285 | 2.411 | 1.207 | 1.359 | 1.244 | Outlier |
| 23 | AJECEX01[r] | 2,6-diamino-pyridin-1-ium | 6.13 | 3.95 | **II** | 0.072<br>0.121 | 2.435<br>2.464 | 1.298<br>1.295 | 1.309<br>1.332 | 1.241<br>1.237 | One of the title molecules has similarly long C═O distances. |
| 24 | AJECIB[s] | 2-amino-pyrimidinium | 6.82 | 4.64 | **II** | 0.114<br>0.145 | 2.466<br>2.473 | 1.277<br>1.270 | 1.308<br>1.323 | 1.241<br>1.238 | |
| 25 | TUMWAB[t] | 1*H*-imidazol-3-ium | 6.95 | 4.77 | **III** | −0.01 | 2.457 | 1.320 | 1.279 | 1.214 | |
| 26 | LUMJOU[u] | hydrazinium | 8.12 | 5.94 | **III** | 0.014 | 2.459 | 1.318 | 1.275 | 1.211 | |
| 27 | SEDKET[v] | 3,5-dimethyl-pyrazolium | 9 | 6.82 | **III** | 0.037 | 2.481 | 1.300 | 1.282 | 1.224 | |
| 28 | SEDKET[v] (corrected) | 3,5-dimethyl-pyrazolium | 9 | 6.82 | **II** | 0.027 | 2.476 | 1.305 | 1.277 | 1.229 | The *bridging hydrogen* after recalculation is closer to oxygen O1, which forms the shorter C═O bond (C12—O1). |
| 29 | LUDDET[w] | benzyl-ammonium | 9.33 | 7.15 | **III** | 0.002 | 2.483 | 1.305 | 1.269 | 1.218 | |
| 30 | LUDDET01[w] | benzyl-ammonium | 9.33 | 7.15 | **III** | | | 1.311<br>1.311 | 1.275<br>1.279 | 1.217<br>1.219 | |

**Table 4 (continued)**

| | Refcode | Base and its form present in the structure | p$K_a$ | Δp$K_a$ | Type | ($q1$ + $q2$) (Å) | $D13$ (Å) | $D1$ (Å) | $D2$ (Å) | $D5$ (Å) | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 31 | INELUI[x] | 1-phenylethyl-ammonium | 9.79 | 7.61 | **III** | 0.009<br>0.009 | 2.467<br>2.482 | 1.309<br>1.320 | 1.272<br>1.277 | 1.221<br>1.214 | |
| 32 | MILLOI[y] | dicyclohexyl-ammonium | 10.4 | 8.22 | **III** | 0.028 | 2.464 | 1.289 | 1.273 | 1.225 | The C═O distances of the title molecule are similar. |
| 33 | ACIFAT[z] | 4-sulfamoyl-anilinium | 10.6 | 8.42 | **III** | 0.028 | 2.462 | 1.315 | 1.287 | 1.209 | |
| 34 | EGUTIJ[aa] | methyl-ammonium | 10.6 | 8.42 | **III** | 0.011 | 2.481 | 1.314 | 1.276 | 1.218 | |
| 35 | EGUTOP[bb] | triethyl-ammonium | 10.78 | 8.6 | **II** | 0.082 | 2.429 | 1.275 | 1.286 | 1.248 | |
| 36 | EGUTOP01[bb] | triethyl-ammonium | 10.78 | 8.6 | **II** | 0.072 | 2.419 | 1.275 | 1.288 | 1.242 | |
| 37 | FOGZIL[cc] | diethyl-ammonium | 11.09 | 8.91 | **III** | 0.004 | 2.489 | 1.308 | 1.270 | 1.217 | |
| 38 | XEBFAM[dd] | piperidinium $C_5H_{11}N$ | 11.28 | 9.1 | **II** and **IV** | 0.078<br>0.061 | 2.586<br>2.736 | 1.219<br>1.234 | 1.278<br>1.253 | 1.255<br>1.271 | One molecule of DNSA (**I**) is fully ionized, the other is in form **II**. |
| 39 | YEJZAO[ee] | guanidinium | 12.5 | 10.32 | **II** | 0.079 | 2.415 | 1.291 | 1.305 | 1.235 | |
| 40 | YEJZAO01[ee] | guanidinium | 12.5 | 10.32 | **II** | 0.073 | 2.415 | 1.292 | 1.300 | 1.239 | |

References for the p$K_a$ values: (*a*) https://chemaxon.com/products/calculators-and-predictors#pka; (*b*) https://www.chemicalbook.com/ProductMSDSDetailCB0688145_EN.htm; (*c*) Dean (1987); (*d*) https://pubchem.ncbi.nlm.nih.gov/compound/nicotinamide#section=p$K_a$; (*e*) https://labs.chem.ucsb.edu/zhang/liming/pdf/pKas_of_Organic_Acids_and_Bases.pdf; (*f*) http://binarystore.wiley.com/store/10.1002/jcc.23068/asset/supinfo/JCC_23068_sm_SuppInfo.pdf?v=1&s=e864a51d58b4cdc175f6b69c92ceddb546201e3b; (*g*) http://sites.chem.colostate.edu/diverdi/all_courses/CRC%20reference%20data/dissociation%20constants%20of%20organic%20acids%20and%20bases.pdf; (*h*) http://sites.chem.colostate.edu/diverdi/all_courses/CRC%20reference%20data/dissociation%20constants%20of%20organic%20acids%20and%20bases.pdf; (*i*) http://chemicalland21.com/specialtychem/finechem/1,10-PHENANTHROLINE.htm; (*j*) https://www.chemicalbook.com/ProductMSDSDetailCB5195697_EN.htm; (*k*) http://www.drugfuture.com/chemdata/cytosine.html; (*l*) https://pubchem.ncbi.nlm.nih.gov/compound/aniline#section=pKa; (*m*) http://sites.chem.colostate.edu/diverdi/all_courses/CRC%20reference%20data/dissociation%20constants%20of%20organic%20acids%20and%20bases.pdf; (*n*) Hosmane & Liebman (2009); (*o*) http://www.csun.edu/\~hcchm003/321/Ka.pdf; (*p*) https://pubchem.ncbi.nlm.nih.gov/compound/pyridine#section=Dissociation-Constants; (*q*) https://onlinelibrary.wiley.com/doi/pdf/10.1002/jcc.23068; (*r*) https://www.chemicalbook.com/ProductMSDSDetailCB0236195_EN.htm; (*s*) https://pubchem.ncbi.nlm.nih.gov/compound/2-aminopyridine#section=Dissociation-Constants; (*t*) https://pubchem.ncbi.nlm.nih.gov/compound/imidazole#section=pKa; (*u*) http://evans.rc.fas.harvard.edu/pdf/evans_p-Ka_table.pdf; (*v*) https://www.chemicalbook.com/ProductMSDSDetailCB2707394_EN.htm; (*w*) https://pubchem.ncbi.nlm.nih.gov/compound/benzylamine#section=p$K_a$; (*x*) https://www.drugbank.ca/drugs/DB04325; (*z*) https://pubchem.ncbi.nlm.nih.gov/compound/dicyclohexylamine#section=Dissociation-Constants; (*z*) https://pubchem.ncbi.nlm.nih.gov/compound/sulfanilamide#section=Dissociation-Constants; (*aa*) https://pubchem.ncbi.nlm.nih.gov/compound/methylamine#section=p$K_a$; (*ab*) https://pubchem.ncbi.nlm.nih.gov/compound/triethylamine#section=Dissociation-Constants; (*ac*) https://pubchem.ncbi.nlm.nih.gov/compound/diethylamine#section=Dissociation-Constants; (*ad*) https://pubchem.ncbi.nlm.nih.gov/compound/piperidine#section=Dissociation-Constants; (*ae*) https://pubchem.ncbi.nlm.nih.gov/compound/guanidine#section=p$K_a$.References to publications with the chemical names of the determined compounds (original and corrected ones if necessary): (1) Senthil Kumar *et al.* (1999): 3,5-dinitrosalicylic acid 1,4-dioxane solvate, 3,5-dinitrosalicylic acid 1,4-dioxane (1:1)]; (2) Senthil Kumar *et al.* (1999): 3,5-dinitrosalicylic acid 1,4-dioxane solvate, 3,5-dinitrosalicylic acid 1,4-dioxane (2:1); (3) Senthil Kumar *et al.* (1999): 3,5-dinitrosalicylic acid 1,4-dioxane solvate, 3,5-dinitrosalicylic acid 1,4-dioxane (2:1); (4) Smith *et al.* (2003*a*): 4-cyanopyridinium 3,5-dinitrosalicylate, 4-cyanopyridinium 3,5-dinitrosalicylate 2-carboxy-4,6-dinitrophenolate; (5) Smith *et al.* (2011): 2-aminoanilinium 2-carboxy-4,6-dinitrophenolate; (6) Khan *et al.* (2013): 2-aminoanilinium 2-hydroxy-3,5-dinitrobenzoate, 2-aminoanilinium 2-carboxy-4,6-dinitrophenolate; (7) Jin *et al.* (2013): 3-carbamoylpyridinium 2-carboxy-4,6-dinitrophenolate, 3-carbamoylpyridinium 2-hydroxy-3,5-dinitrobenzoate; (8) Smith *et al.* (2007): 1-naphthylammonium 3,5-dinitrosalicylate, 1-naphthylammonium 2-carboxy-4,6-dinitrophenolate; (9) Smith *et al.* (2001*b*): 8-aminoquinolinium 3,5-dinitrosalicylate; (10) Smith *et al.* (2011): 4-chloroanilinium 2-hydroxy-3,5-dinitrobenzoate; (11) Jones *et al.* (2014): (4-iodoanilinium 2-hydroxy-3,5-dinitrobenzoate; (12) Smith *et al.* (2007): 1,10-Phenanthrolinium 3,5-dinitrosalicylate; (13) Singh *et al.* (2014): 2-(pyridin-2-yl)pyridinium 2-hydroxy-3,5-dinitrobenzoate; (14) Song *et al.* (2007): 2,2′-bipyridinium 2-carboxy-4,6-dinitrophenolate; (15) Smith *et al.* (2007): 2,2′-bipyridinium 2-carboxy-4,6-dinitrophenolate; (16) Smith *et al.* (2005*a*): cytosinium 3,5-dinitrosalicylate, cytosinium 2-carboxy-4,6-dinitrophenolate; (17) Smith *et al.* (2011): anilinium 2-hydroxy-3,5-dinitrobenzoate; (18) Smith *et al.* (2011): 4-fluoroanilinium 2-hydroxy-3,5-dinitrobenzoate; (19) Smith *et al.* (2007): quinolinium 3,5-dinitrosalicylate, quinolinium 2-carboxy-4,6-dinitrophenolate; (20) Smith *et al.* (1995): 3,5-dinitrosalicylic acid 2-aminobenzoic acid, 2-ammoniumbenzoic acid 2-carboxy-4,6-dinitrophenolate; (21) Smith *et al.* (2003*a*): pyridinium 3,5-dinitrosalicylate 3,5-dinitrosalicylic acid; (22) Zhang *et al.* (2014): 2-methylquinolinium 2-hydroxy-3,5-dinitrobenzoate; (23) Gao *et al.* (2015): 2,6-diaminopyridin-1-ium 2-hydroxy-3,5-dinitrobenzoate; (24) Smith *et al.* (2003*a*): 2-aminopyrimidinium 3,5-dinitrosalicylate ethanol solvate, 2-aminopyrimidinium 3,5-dinitrosalicylate ethanol (2:2:1); (25) Jin *et al.* (2015*b*): 1*H*-imidazol-3-ium 2-carboxy-4,6-dinitrophenolate; (26) Fu *et al.* (2015): hydrazinium 2-carboxy-4,6-dinitrophenolate; (27) Wei *et al.* (2012): (3,5-Dimethylpyrazolium 2-carboxy-4,6-dinitrophenolate); (28) this work: (3,5-dimethylpyrazolium 2-hydroxy-3,5-dinitrobenzoate; (29) Smith *et al.* (2002*b*): benzylammonium 3,5-dinitrosalicylate, benzylammonium 2-carboxy-4,6-dinitrophenolate; (30) Jin *et al.* (2015*a*): benzylammonium 2-carboxy-4,6-dinitrophenolate; (31) Smith *et al.* (2003*b*): (*S*)-(−)-1-phenylethylaminium 3,5-dinitrosalicylate, (*S*)-(−)-1-phenylethylaminium 2-carboxy-4,6-dinitrophenolate; (32) Ng *et al.* (2001): dicyclohexylammonium 2-carboxy-4,6-dinitrophenolate; (33) Smith *et al.* (2001*c*): 4-ammoniobenzenesulfonamide 3,5-dinitrosalicylate, 4-ammoniobenzenesulfonamide 2-carboxy-4,6-dinitrophenolate; (34) Smith *et al.* (2002*a*): methylammonium 3,5-dinitrosalicylate, methylammonium 2-carboxy-4,6-dinitrophenolate; (35) Smith *et al.* (2002*a*): triethylammonium 3,5-dinitrosalicylate; (36) Rajkumar & Chandramohan (2017): triethylammonium 2-hydroxy-3,5-dinitrobenzoate; (37) Smith *et al.* (2005*b*): diethylammonium 3,5-dinitrosalicylate, diethylammonium 2-carboxy-4,6-dinitrophenolate; (38) Smith *et al.* (2006): tris(piperidinium) bis(3,5-dinitrosalicylate) monohydrate, tris(piperidinium) 2-hydroxy-3,5-dinitrobenzoate 2-olate-3,5-dinitrobenzoate monohydrate; (39) Smith *et al.* (2001*a*): guanidinium 3,5-dinitrosalicylate; (40) Fu *et al.* (2015): guanidinium 3,5-dinitrosalicylate.

3,5-dinitrobenzoate (**II**) or 2-carboxy-4,6-dinitrophenolate (**III**). These structures appear in the intermediate region of Table 4. A more radical transfer of the acid hydrogen atom should cause a more significant shortening of bond $D5$, which should be concomitant with the elongation of bond $D1$. Such an elongation of bond $D1$ (*cf.* Fig. 1*a*) should support the formation of a 2-carboxy-4,6-dinitrophenolate (**III**).

## 4. Summary

(1) The *bridging hydrogen* in the molecules discussed (**I**–**III**) is involved in a resonance-assisted hydrogen bond, which is part of a hexagonal $R_1^1(6)$ ring. The system of conjugated bonds in the title molecules, however, comprises more atoms than the ring in which the *bridging hydrogen* is involved. In particular, the whole carboxylate/carboxylic group affects the discussed intramolecular O···H···O hydrogen bond.

(2) The transition region between the forms of 2-hydroxy-3,5-dinitrobenzoates (**II**) and 2-carboxy-4,6-dinitrophenolates (**III**) takes place for C—O ($D1$) ≃ 1.28–1.30 Å, C—O ($D2$) ≃ 1.30 Å, O···O distance $D13$ ≃ 2.41 Å and ($q1$ + $q2$) ≃ 0.08 Å. Simultaneously, the highest probability for the presence of the *bridging hydrogen* to be in the centre of the hydrogen bond is expected in this transition region. However, the hydrogen

atom can also be disordered over two positions as occurs in NUQVEB.

(3) The *bridging hydrogen* in the discussed intramolecular hydrogen bond can be situated at the centre between both oxygen atoms with approximately equal C—O bond distances. Therefore, the *bridging hydrogen* can not be situated at the centre of the intramolecular O···H···O hydrogen bond in compounds containing 2-hydroxy-3,5-dinitrobenzoic acid (**I**).

(4) In some rare cases (*e.g.* recalculated SEDKET, KEZJIJ and KEZJIJ01), the *bridging hydrogen* is bonded to the oxygen atom that forms the shorter C—O bond distance (Table 3). It would be of interest to see how the localization of the *bridging hydrogen* develops with changing temperature in such cases.

(5) Table 4 shows the occurrence of the different forms of the molecules (see scheme) and the dependence on basicity. Alhough it would be expected that the increasing basicity should support the occurrence of 2-carboxy-4,6-dinitrophenolates (**III**) and, of course, for very strong bases, 3,5-dinitro-2-oxidobenzoates (**IV**), there are many exceptions to this rule.

(6) The positioning of the hydrogen atoms can be affected by the asphericity of the electron density of the donor and acceptor atoms.

(7) It is essential to calculate difference electron-density maps in order to locate correctly the *bridging hydrogen* atom, and any other hydrogen atoms involved in hydrogen bonding.

(8) The present overview has shown that the application of constraints and restraints is frequently incorrect.

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# supporting information

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## A resonance-assisted intramolecular hydrogen bond in compounds containing 2-hydroxy-3,5-dinitrobenzoic acid and its various deprotonated forms: redetermination of several related structures

**Jan Fábry**

### Computing details

Data collection: *SMART* (Bruker, 1997) for DUJZAK; *SMART* (Bruker, 1999) for JEVNAA; *CAD-4 Software* (Enraf-Nonius, 1989) for LUDFUL; *APEX2* (Bruker, 2009) for NUQVEB, VABZIJ; *SMART* (Bruker, 2001) for QIQJAD; *SMART* (Bruker, 2007) for SAFGUD; *SMART* (Bruker, 2002) for SEDKET; *CrysAlis PRO* (Agilent, 2012) for TIYZIM; *APEX2* (Bruker, 2004) for TUJPEV; *CrysAlis PRO* (Agilent, 2014) for WADXOR; *APEX2* (Bruker, 2010) for YAXPOE. Cell refinement: *SAINT* (Bruker, 1997) for DUJZAK; *SAINT* (Bruker, 1999) for JEVNAA; *CAD-4 Software* (Enraf-Nonius, 1989) for LUDFUL; *SAINT* (Bruker, 2009) for NUQVEB, VABZIJ; *SAINT* (Bruker, 2001) for QIQJAD; *SAINT* (Bruker, 2007) for SAFGUD; *SAINT* (Bruker, 2002) for SEDKET; *CrysAlis PRO* (Agilent, 2012) for TIYZIM; *SAINT* (Bruker, 2004) for TUJPEV; *CrysAlis PRO* (Agilent, 2014) for WADXOR; *SAINT* (Bruker, 2010) for YAXPOE. Data reduction: *SAINT* (Bruker, 1997) for DUJZAK; *SAINT* (Bruker, 1999) for JEVNAA; *Xtal3*.5 (Hall *et al.*, 1995) for LUDFUL; *SAINT* (Bruker, 2009) for NUQVEB, VABZIJ; *SAINT* (Bruker, 2001) for QIQJAD; *SAINT* (Bruker, 2007) for SAFGUD; *SAINT* (Bruker, 2002) for SEDKET; *CrysAlis RED* (Agilent, 2012) for TIYZIM; *SAINT* (Bruker, 2004) for TUJPEV; *CrysAlis PRO* (Agilent, 2014) for WADXOR; *SAINT* (Bruker, 2010) for YAXPOE. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008) for DUJZAK, JEVNAA, LUDFUL, QIQJAD, SEDKET, TUJPEV, YAXPOE; *SHELXTL* (Sheldrick, 2008) for NUQVEB, VABZIJ; *SHELXS-97* (Sheldrick, 2008) for SAFGUD; SUPERFLIP (Palatinus & Chapuis, 2007) for TIYZIM; *SIR92* (Altomare *et al.*, 1993) for WADXOR. Program(s) used to refine structure: JANA2016 (Petricek *et al.*, 2014) for DUJZAK; JANA2006 (Petricek *et al.*, 2014) for JEVNAA, LUDFUL, NUQVEB, QIQJAD, SAFGUD, TIYZIM, TUJPEV, VABZIJ, YAXPOE; JANA2006 (Petricek, 2014) for SEDKET.

### Bis(quinolin-8-ol)silver(I) 2-hydroxy-3,5-dinitrobenzoate (DUJZAK)

*Crystal data*

$[Ag(C_9H_7NO)_2](C_7H_3N_2O_7)$
$M_r$ = 625.30
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a$ = 9.0154 (18) Å
$b$ = 7.6122 (15) Å
$c$ = 17.138 (3) Å
$\beta$ = 104.38 (3)°
$V$ = 1139.3 (4) Å$^3$
$Z$ = 2
$F(000)$ = 628
$D_x$ = 1.823 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4356 reflections
$\theta$ = 3.6–27.6°
$\mu$ = 0.95 mm$^{-1}$
$T$ = 293 K
Block, yellow
0.20 × 0.15 × 0.11 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
10841 measured reflections
4602 independent reflections
4225 reflections with $I > 3\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.6°$
$h = -11 \rightarrow 11$
$k = -9 \rightarrow 8$
$l = -22 \rightarrow 22$

*Refinement*

Refinement on $F^2$
$R[F > 3\sigma(F)] = 0.023$
$wR(F) = 0.053$
$S = 1.34$
4602 reflections
356 parameters
0 restraints
48 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max} = 0.025$
$\Delta\rho_{max} = 0.44$ e Å$^{-3}$
$\Delta\rho_{min} = -0.30$ e Å$^{-3}$
Absolute structure: 1800 of Friedel pairs used in the refinement
Absolute structure parameter: 0.004 (17)

*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**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.
Number of fixed parameters 9.

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Ag1 | 0.062197 (18) | 0.74282 (3) | 0.668863 (11) | 0.01868 (5) |
| O1 | −0.0993 (2) | 0.4630 (2) | 0.64021 (12) | 0.0202 (6) |
| O2 | 0.1741 (2) | 0.5061 (2) | 0.77196 (12) | 0.0214 (6) |
| N1 | −0.1154 (2) | 0.7667 (3) | 0.55616 (13) | 0.0177 (7) |
| N2 | 0.2685 (2) | 0.8371 (3) | 0.75427 (14) | 0.0163 (7) |
| C1 | −0.1280 (3) | 0.9181 (4) | 0.51463 (18) | 0.0222 (9) |
| H1a | −0.061241 | 1.009276 | 0.535386 | 0.0267* |
| C2 | −0.2362 (3) | 0.9448 (4) | 0.44205 (18) | 0.0243 (9) |
| H2a | −0.240923 | 1.051917 | 0.415439 | 0.0291* |
| C3 | −0.3354 (3) | 0.8130 (4) | 0.41016 (19) | 0.0209 (9) |
| H3a | −0.407848 | 0.829471 | 0.361612 | 0.0251* |
| C4 | −0.3270 (3) | 0.6506 (4) | 0.45167 (18) | 0.0169 (8) |
| C5 | −0.4264 (3) | 0.5079 (4) | 0.42259 (17) | 0.0209 (9) |
| H5a | −0.500744 | 0.518344 | 0.37429 | 0.0251* |
| C6 | −0.4131 (3) | 0.3554 (4) | 0.46539 (17) | 0.0217 (9) |

| | | | | |
|---|---|---|---|---|
| H6a | −0.477402 | 0.261662 | 0.445311 | 0.026* |
| C7 | −0.3036 (3) | 0.3371 (3) | 0.53948 (17) | 0.0184 (8) |
| H7a | −0.297965 | 0.232672 | 0.568204 | 0.0221* |
| C8 | −0.2057 (3) | 0.4717 (3) | 0.56954 (16) | 0.0148 (8) |
| C9 | −0.2139 (3) | 0.6331 (3) | 0.52566 (16) | 0.0141 (8) |
| C10 | 0.3159 (3) | 0.9993 (4) | 0.74633 (17) | 0.0188 (9) |
| H10a | 0.256892 | 1.069872 | 0.706108 | 0.0225* |
| C11 | 0.4512 (3) | 1.0703 (4) | 0.79567 (18) | 0.0220 (9) |
| H11a | 0.480885 | 1.184529 | 0.787703 | 0.0264* |
| C12 | 0.5373 (3) | 0.9693 (4) | 0.85495 (18) | 0.0210 (9) |
| H12a | 0.627346 | 1.01397 | 0.887796 | 0.0252* |
| C13 | 0.4907 (3) | 0.7960 (3) | 0.86712 (16) | 0.0171 (8) |
| C14 | 0.5743 (3) | 0.6845 (4) | 0.92853 (17) | 0.0200 (8) |
| H14a | 0.665083 | 0.723938 | 0.962749 | 0.024* |
| C15 | 0.5225 (3) | 0.5197 (4) | 0.93771 (17) | 0.0206 (8) |
| H15a | 0.577789 | 0.447807 | 0.978539 | 0.0247* |
| C16 | 0.3863 (3) | 0.4570 (3) | 0.88620 (16) | 0.0173 (8) |
| H16a | 0.35178 | 0.344669 | 0.8937 | 0.0208* |
| C17 | 0.3043 (3) | 0.5596 (3) | 0.82528 (16) | 0.0142 (8) |
| C18 | 0.3541 (2) | 0.7340 (5) | 0.81443 (13) | 0.0140 (6) |
| O3 | 0.1402 (2) | −0.0134 (3) | 0.92525 (13) | 0.0259 (7) |
| O4 | −0.4152 (2) | −0.3755 (3) | 0.69139 (13) | 0.0284 (7) |
| O5 | −0.3271 (2) | −0.5961 (3) | 0.76858 (13) | 0.0271 (7) |
| O6 | 0.1238 (2) | −0.5247 (2) | 0.98352 (12) | 0.0211 (6) |
| O7 | 0.1679 (2) | −0.2659 (4) | 1.03546 (11) | 0.0286 (6) |
| O8 | −0.0981 (2) | 0.1498 (2) | 0.70180 (12) | 0.0237 (7) |
| O9 | 0.06657 (19) | 0.2138 (2) | 0.81885 (11) | 0.0196 (6) |
| N3 | −0.3217 (2) | −0.4436 (3) | 0.74764 (14) | 0.0173 (7) |
| N4 | 0.1088 (2) | −0.3645 (3) | 0.98085 (13) | 0.0152 (7) |
| C19 | −0.1970 (3) | −0.3339 (4) | 0.79254 (18) | 0.0130 (8) |
| C20 | −0.1027 (3) | −0.3992 (3) | 0.86303 (15) | 0.0126 (7) |
| H20a | −0.114934 | −0.512855 | 0.880295 | 0.0151* |
| C21 | 0.0095 (2) | −0.2908 (3) | 0.90660 (15) | 0.0114 (8) |
| C22 | 0.0326 (3) | −0.1188 (3) | 0.88194 (16) | 0.0130 (8) |
| C23 | −0.0605 (3) | −0.0603 (3) | 0.80708 (16) | 0.0129 (7) |
| C24 | −0.1770 (3) | −0.1678 (3) | 0.76332 (17) | 0.0132 (8) |
| H24a | −0.240539 | −0.128661 | 0.715089 | 0.0159* |
| C25 | −0.0307 (3) | 0.1145 (3) | 0.77245 (16) | 0.0148 (8) |
| H1aa | −0.091407 | 0.377732 | 0.663815 | 0.047 (14)* |
| H2aa | 0.145536 | 0.395004 | 0.783901 | 0.043 (11)* |
| H3b | 0.135279 | 0.095353 | 0.887993 | 0.17 (3)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ag1 | 0.01622 (8) | 0.01722 (9) | 0.01945 (10) | −0.00339 (11) | −0.00154 (6) | 0.00024 (11) |
| O1 | 0.0219 (9) | 0.0127 (9) | 0.0199 (10) | −0.0063 (8) | −0.0065 (8) | 0.0058 (8) |
| O2 | 0.0185 (9) | 0.0173 (9) | 0.0231 (11) | −0.0065 (8) | −0.0049 (8) | 0.0030 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.0190 (9) | 0.0152 (14) | 0.0181 (11) | −0.0003 (11) | 0.0033 (8) | 0.0008 (10) |
| N2 | 0.0145 (10) | 0.0161 (11) | 0.0187 (12) | −0.0018 (9) | 0.0049 (9) | 0.0005 (9) |
| C1 | 0.0254 (14) | 0.0145 (12) | 0.0263 (16) | −0.0042 (12) | 0.0056 (12) | 0.0044 (11) |
| C2 | 0.0325 (15) | 0.0177 (13) | 0.0239 (16) | 0.0069 (13) | 0.0093 (12) | 0.0104 (11) |
| C3 | 0.0214 (13) | 0.0247 (13) | 0.0154 (15) | 0.0059 (12) | 0.0021 (12) | 0.0023 (11) |
| C4 | 0.0136 (12) | 0.0220 (14) | 0.0150 (15) | 0.0026 (11) | 0.0031 (10) | 0.0005 (12) |
| C5 | 0.0160 (12) | 0.0289 (15) | 0.0156 (14) | 0.0005 (12) | −0.0002 (10) | −0.0035 (12) |
| C6 | 0.0156 (12) | 0.0247 (14) | 0.0217 (16) | −0.0092 (12) | −0.0009 (11) | −0.0062 (11) |
| C7 | 0.0179 (12) | 0.0175 (14) | 0.0187 (14) | −0.0039 (11) | 0.0026 (10) | 0.0018 (11) |
| C8 | 0.0139 (12) | 0.0146 (12) | 0.0140 (13) | 0.0001 (11) | 0.0000 (9) | −0.0001 (10) |
| C9 | 0.0147 (12) | 0.0142 (12) | 0.0132 (13) | −0.0002 (11) | 0.0032 (9) | 0.0016 (10) |
| C10 | 0.0208 (13) | 0.0176 (13) | 0.0198 (15) | −0.0021 (12) | 0.0084 (11) | 0.0021 (11) |
| C11 | 0.0259 (14) | 0.0166 (13) | 0.0254 (16) | −0.0085 (12) | 0.0099 (12) | −0.0039 (11) |
| C12 | 0.0180 (12) | 0.0226 (14) | 0.0235 (15) | −0.0108 (12) | 0.0074 (11) | −0.0101 (12) |
| C13 | 0.0138 (11) | 0.0232 (14) | 0.0156 (14) | −0.0032 (10) | 0.0064 (10) | −0.0059 (10) |
| C14 | 0.0118 (11) | 0.0285 (14) | 0.0182 (15) | −0.0034 (11) | 0.0010 (10) | −0.0071 (10) |
| C15 | 0.0143 (12) | 0.0281 (15) | 0.0175 (15) | 0.0038 (12) | 0.0002 (10) | 0.0017 (11) |
| C16 | 0.0160 (12) | 0.0146 (12) | 0.0208 (15) | −0.0018 (11) | 0.0035 (10) | −0.0001 (10) |
| C17 | 0.0107 (11) | 0.0149 (12) | 0.0165 (14) | −0.0020 (10) | 0.0025 (9) | −0.0031 (10) |
| C18 | 0.0115 (9) | 0.0160 (11) | 0.0156 (11) | 0.0014 (17) | 0.0052 (8) | 0.0001 (15) |
| O3 | 0.0215 (10) | 0.0236 (11) | 0.0279 (12) | −0.0066 (9) | −0.0029 (9) | 0.0016 (9) |
| O4 | 0.0212 (10) | 0.0283 (11) | 0.0267 (12) | −0.0044 (9) | −0.0110 (8) | −0.0010 (9) |
| O5 | 0.0295 (10) | 0.0195 (10) | 0.0288 (12) | −0.0130 (9) | 0.0004 (9) | 0.0016 (9) |
| O6 | 0.0213 (9) | 0.0158 (9) | 0.0236 (11) | 0.0028 (8) | 0.0007 (8) | 0.0059 (8) |
| O7 | 0.0340 (9) | 0.0225 (9) | 0.0194 (9) | 0.0057 (15) | −0.0121 (7) | −0.0015 (13) |
| O8 | 0.0304 (11) | 0.0156 (10) | 0.0204 (11) | −0.0061 (9) | −0.0028 (8) | 0.0051 (8) |
| O9 | 0.0209 (8) | 0.0129 (12) | 0.0223 (10) | −0.0061 (8) | 0.0003 (7) | 0.0009 (8) |
| N3 | 0.0141 (10) | 0.0190 (11) | 0.0169 (12) | −0.0064 (10) | 0.0001 (9) | −0.0044 (9) |
| N4 | 0.0115 (10) | 0.0169 (11) | 0.0154 (12) | 0.0012 (9) | −0.0003 (8) | 0.0025 (9) |
| C19 | 0.0080 (11) | 0.0164 (12) | 0.0135 (15) | −0.0043 (10) | 0.0009 (10) | −0.0044 (11) |
| C20 | 0.0158 (12) | 0.0082 (11) | 0.0137 (13) | −0.0009 (10) | 0.0036 (9) | 0.0004 (9) |
| C21 | 0.0098 (9) | 0.0122 (17) | 0.0103 (11) | 0.0048 (10) | −0.0012 (8) | 0.0021 (9) |
| C22 | 0.0086 (11) | 0.0150 (13) | 0.0147 (14) | −0.0009 (10) | 0.0015 (9) | −0.0033 (10) |
| C23 | 0.0133 (11) | 0.0110 (12) | 0.0134 (13) | −0.0001 (10) | 0.0015 (9) | −0.0007 (9) |
| C24 | 0.0120 (12) | 0.0135 (13) | 0.0142 (15) | 0.0018 (11) | 0.0033 (10) | 0.0004 (11) |
| C25 | 0.0153 (12) | 0.0109 (11) | 0.0177 (14) | 0.0006 (10) | 0.0029 (10) | 0.0010 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.347 (3) | C13—C14 | 1.415 (4) |
| O1—H1aa | 0.7585 (19) | C13—C18 | 1.415 (3) |
| O2—C17 | 1.359 (3) | C14—H14a | 0.93 |
| O2—H2aa | 0.922 (2) | C14—C15 | 1.361 (4) |
| N1—C1 | 1.344 (4) | C15—H15a | 0.93 |
| N1—C9 | 1.365 (3) | C15—C16 | 1.406 (3) |
| N2—C10 | 1.325 (4) | C16—H16a | 0.93 |
| N2—C18 | 1.371 (4) | C16—C17 | 1.365 (4) |
| C1—H1a | 0.93 | C17—C18 | 1.429 (5) |

| | | | |
|---|---|---|---|
| C1—C2 | 1.392 (4) | O3—C22 | 1.333 (3) |
| C2—H2a | 0.93 | O3—H3b | 1.040 (2) |
| C2—C3 | 1.364 (4) | O4—N3 | 1.227 (3) |
| C3—H3a | 0.93 | O5—N3 | 1.219 (3) |
| C3—C4 | 1.419 (4) | O6—N4 | 1.226 (3) |
| C4—C5 | 1.418 (4) | O7—N4 | 1.215 (3) |
| C4—C9 | 1.423 (4) | O8—C25 | 1.242 (3) |
| C5—H5a | 0.93 | O9—C25 | 1.275 (3) |
| C5—C6 | 1.362 (4) | O9—H3b | 1.4952 (19) |
| C6—H6a | 0.93 | N3—C19 | 1.458 (3) |
| C6—C7 | 1.408 (4) | N4—C21 | 1.473 (3) |
| C7—H7a | 0.93 | C19—C20 | 1.385 (4) |
| C7—C8 | 1.367 (4) | C19—C24 | 1.388 (4) |
| C8—C9 | 1.433 (4) | C20—H20a | 0.93 |
| C10—H10a | 0.93 | C20—C21 | 1.373 (3) |
| C10—C11 | 1.408 (4) | C21—C22 | 1.408 (4) |
| C11—H11a | 0.93 | C22—C23 | 1.419 (3) |
| C11—C12 | 1.354 (4) | C23—C24 | 1.394 (3) |
| C12—H12a | 0.93 | C23—C25 | 1.508 (4) |
| C12—C13 | 1.416 (4) | C24—H24a | 0.93 |

| | | | |
|---|---|---|---|
| C8—O1—H1aa | 118.2 (2) | C13—C14—C15 | 120.2 (2) |
| C17—O2—H2aa | 111.60 (19) | H14a—C14—C15 | 119.88 |
| C1—N1—C9 | 118.3 (2) | C14—C15—H15a | 119.61 |
| C10—N2—C18 | 118.4 (2) | C14—C15—C16 | 120.8 (2) |
| N1—C1—H1a | 118.43 | H15a—C15—C16 | 119.61 |
| N1—C1—C2 | 123.1 (2) | C15—C16—H16a | 119.71 |
| H1a—C1—C2 | 118.43 | C15—C16—C17 | 120.6 (2) |
| C1—C2—H2a | 120.19 | H16a—C16—C17 | 119.71 |
| C1—C2—C3 | 119.6 (3) | O2—C17—C16 | 123.8 (2) |
| H2a—C2—C3 | 120.19 | O2—C17—C18 | 116.0 (2) |
| C2—C3—H3a | 120.26 | C16—C17—C18 | 120.2 (2) |
| C2—C3—C4 | 119.5 (2) | N2—C18—C13 | 121.8 (3) |
| H3a—C3—C4 | 120.27 | N2—C18—C17 | 119.6 (2) |
| C3—C4—C5 | 122.8 (2) | C13—C18—C17 | 118.6 (2) |
| C3—C4—C9 | 117.8 (2) | C22—O3—H3b | 102.89 (19) |
| C5—C4—C9 | 119.5 (2) | C25—O9—H3b | 102.84 (17) |
| C4—C5—H5a | 120 | O4—N3—O5 | 124.3 (2) |
| C4—C5—C6 | 120.0 (2) | O4—N3—C19 | 117.5 (2) |
| H5a—C5—C6 | 120 | O5—N3—C19 | 118.2 (2) |
| C5—C6—H6a | 119.4 | O6—N4—O7 | 124.2 (2) |
| C5—C6—C7 | 121.2 (2) | O6—N4—C21 | 116.66 (19) |
| H6a—C6—C7 | 119.4 | O7—N4—C21 | 119.1 (2) |
| C6—C7—H7a | 119.72 | N3—C19—C20 | 118.7 (2) |
| C6—C7—C8 | 120.6 (2) | N3—C19—C24 | 118.9 (2) |
| H7a—C7—C8 | 119.72 | C20—C19—C24 | 122.3 (2) |
| O1—C8—C7 | 123.5 (2) | C19—C20—H20a | 121.04 |
| O1—C8—C9 | 116.5 (2) | C19—C20—C21 | 117.9 (2) |

| | | | |
|---|---|---|---|
| C7—C8—C9 | 120.0 (2) | H20a—C20—C21 | 121.04 |
| N1—C9—C4 | 121.7 (2) | N4—C21—C20 | 116.7 (2) |
| N1—C9—C8 | 119.6 (2) | N4—C21—C22 | 120.67 (19) |
| C4—C9—C8 | 118.7 (2) | C20—C21—C22 | 122.7 (2) |
| N2—C10—H10a | 118.33 | O3—C22—C21 | 122.3 (2) |
| N2—C10—C11 | 123.3 (2) | O3—C22—C23 | 120.0 (2) |
| H10a—C10—C11 | 118.33 | C21—C22—C23 | 117.7 (2) |
| C10—C11—H11a | 120.58 | C22—C23—C24 | 120.0 (2) |
| C10—C11—C12 | 118.8 (3) | C22—C23—C25 | 120.6 (2) |
| H11a—C11—C12 | 120.58 | C24—C23—C25 | 119.4 (2) |
| C11—C12—H12a | 119.87 | C19—C24—C23 | 119.2 (2) |
| C11—C12—C13 | 120.3 (2) | C19—C24—H24a | 120.38 |
| H12a—C12—C13 | 119.87 | C23—C24—H24a | 120.38 |
| C12—C13—C14 | 123.1 (2) | O8—C25—O9 | 125.0 (2) |
| C12—C13—C18 | 117.4 (2) | O8—C25—C23 | 118.9 (2) |
| C14—C13—C18 | 119.6 (3) | O9—C25—C23 | 116.1 (2) |
| C13—C14—H14a | 119.88 | O3—H3b—O9 | 155.88 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C11—H11*a*···O5$^{i}$ | 0.93 | 2.48 | 3.335 (4) | 152 |
| O1—H1*aa*···O8 | 0.7585 (19) | 1.859 (2) | 2.606 (3) | 167.96 (14) |
| O2—H2*aa*···O9 | 0.922 (2) | 1.727 (2) | 2.631 (3) | 166.48 (15) |
| O3—H3*b*···O9 | 1.040 (2) | 1.4952 (19) | 2.481 (3) | 155.88 (12) |

Symmetry code: (i) *x*+1, *y*+2, *z*.

## Tetrakis(1*H*-imidazole-$\kappa N^3$)zinc(II) bis(2-hydroxy-3,5-dinitrobenzoate) (JEVNAA)

*Crystal data*

$[Zn(C_3H_4N_2)_4](C_7H_3N_2O_7)_2$
$M_r$ = 791.93
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
*a* = 25.0809 (15) Å
*b* = 6.7251 (4) Å
*c* = 18.9145 (10) Å
*β* = 97.658 (6)°
*V* = 3161.9 (3) Å$^3$
*Z* = 4
*F*(000) = 1616
$D_x$ = 1.664 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3242 reflections
*θ* = 2.1–26.9°
*μ* = 0.87 mm$^{-1}$
*T* = 293 K
Platelet, yellow
0.20 × 0.18 × 0.10 mm

*Data collection*

Bruker APEX-II area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*φ* and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 1999)
$T_{min}$ = 0.846, $T_{max}$ = 0.918
20634 measured reflections
3635 independent reflections
2152 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.058
$\theta_{max}$ = 27.6°, $\theta_{min}$ = 1.6°
*h* = −32→31
*k* = −8→8
*l* = −24→24

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)] = 0.036$
$wR(F) = 0.075$
$S = 1.23$
3635 reflections
244 parameters
0 restraints
32 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{min} = -0.23$ e Å$^{-3}$
Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 1400 (500)

*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**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
Number of fixed parameters: 9

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.47960 (10) | 0.4200 (3) | 0.62639 (13) | 0.0460 (10) |
| H1 | 0.444004 | 0.398202 | 0.633096 | 0.0552* |
| C2 | 0.50386 (10) | 0.3369 (4) | 0.57533 (13) | 0.0484 (10) |
| H2 | 0.488745 | 0.248295 | 0.540565 | 0.0581* |
| C3 | 0.56018 (9) | 0.5299 (3) | 0.63970 (12) | 0.0401 (9) |
| H3 | 0.591591 | 0.597819 | 0.656666 | 0.0481* |
| C4 | 0.59325 (9) | 0.9065 (4) | 0.83991 (12) | 0.0404 (9) |
| H4 | 0.591722 | 0.823229 | 0.87885 | 0.0485* |
| N4 | 0.62879 (7) | 1.0508 (3) | 0.83789 (11) | 0.0471 (8) |
| C6 | 0.57665 (9) | 1.0451 (4) | 0.73735 (13) | 0.0444 (9) |
| H6 | 0.560952 | 1.074705 | 0.691199 | 0.0533* |
| C7 | 0.73999 (9) | 0.4553 (3) | 0.34225 (11) | 0.0329 (8) |
| C8 | 0.79245 (9) | 0.5059 (3) | 0.36676 (12) | 0.0342 (8) |
| H8 | 0.817639 | 0.51762 | 0.335092 | 0.0411* |
| C9 | 0.80717 (8) | 0.5388 (3) | 0.43803 (12) | 0.0311 (8) |
| C10 | 0.77048 (8) | 0.5210 (3) | 0.48978 (12) | 0.0302 (8) |
| C11 | 0.71702 (8) | 0.4593 (3) | 0.46099 (11) | 0.0282 (7) |
| C12 | 0.70286 (9) | 0.4300 (3) | 0.38898 (12) | 0.0323 (8) |
| H12 | 0.667845 | 0.392676 | 0.371656 | 0.0388* |
| C13 | 0.67545 (9) | 0.4249 (3) | 0.50834 (12) | 0.0333 (8) |
| N1 | 0.51487 (7) | 0.5429 (3) | 0.66784 (9) | 0.0373 (7) |
| N2 | 0.55523 (8) | 0.4075 (3) | 0.58408 (10) | 0.0438 (8) |

| | | | | |
|---|---|---|---|---|
| N3 | 0.56024 (7) | 0.8957 (3) | 0.77961 (9) | 0.0354 (7) |
| C5 | 0.61854 (9) | 1.1406 (4) | 0.77303 (14) | 0.0479 (10) |
| H5 | 0.637064 | 1.247402 | 0.75675 | 0.0575* |
| N5 | 0.72386 (8) | 0.4225 (3) | 0.26644 (10) | 0.0427 (8) |
| N6 | 0.86351 (7) | 0.5897 (3) | 0.46068 (11) | 0.0403 (8) |
| O1 | 0.78238 (6) | 0.5518 (2) | 0.55681 (8) | 0.0378 (6) |
| O2 | 0.68832 (6) | 0.4695 (2) | 0.57640 (8) | 0.0420 (6) |
| O3 | 0.63104 (6) | 0.3579 (2) | 0.48669 (8) | 0.0426 (6) |
| O4 | 0.67630 (7) | 0.3835 (3) | 0.24643 (8) | 0.0575 (7) |
| O5 | 0.75772 (7) | 0.4340 (3) | 0.22570 (9) | 0.0662 (8) |
| O6 | 0.89634 (7) | 0.5475 (3) | 0.42013 (9) | 0.0658 (8) |
| O7 | 0.87622 (6) | 0.6730 (3) | 0.51757 (9) | 0.0568 (7) |
| Zn1 | 0.5 | 0.70908 (6) | 0.75 | 0.03866 (15) |
| H1a | 0.728641 | 0.508268 | 0.579335 | 0.108 (11)* |
| H2a | 0.580668 | 0.367714 | 0.552562 | 0.088 (9)* |
| H4a | 0.657593 | 1.098579 | 0.870594 | 0.106 (11)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0323 (14) | 0.0558 (17) | 0.0507 (17) | −0.0069 (12) | 0.0086 (12) | −0.0071 (13) |
| C2 | 0.0460 (16) | 0.0536 (18) | 0.0448 (17) | −0.0064 (13) | 0.0033 (12) | −0.0116 (13) |
| C3 | 0.0327 (13) | 0.0485 (16) | 0.0396 (16) | −0.0053 (11) | 0.0071 (11) | 0.0037 (12) |
| C4 | 0.0366 (14) | 0.0491 (16) | 0.0343 (15) | 0.0066 (12) | 0.0004 (11) | 0.0062 (12) |
| N4 | 0.0332 (12) | 0.0568 (15) | 0.0484 (15) | −0.0016 (10) | −0.0048 (10) | −0.0044 (11) |
| C6 | 0.0416 (15) | 0.0568 (17) | 0.0336 (15) | −0.0015 (12) | 0.0004 (12) | 0.0077 (13) |
| C7 | 0.0344 (13) | 0.0369 (14) | 0.0264 (14) | 0.0024 (10) | 0.0002 (10) | −0.0008 (10) |
| C8 | 0.0332 (13) | 0.0366 (14) | 0.0337 (15) | 0.0009 (10) | 0.0073 (10) | 0.0030 (10) |
| C9 | 0.0254 (12) | 0.0287 (13) | 0.0382 (15) | −0.0031 (9) | 0.0003 (10) | −0.0001 (10) |
| C10 | 0.0338 (13) | 0.0219 (12) | 0.0342 (14) | 0.0035 (9) | 0.0013 (10) | 0.0006 (10) |
| C11 | 0.0288 (12) | 0.0245 (12) | 0.0310 (14) | 0.0011 (9) | 0.0030 (10) | −0.0013 (10) |
| C12 | 0.0281 (12) | 0.0306 (14) | 0.0370 (15) | 0.0009 (9) | −0.0002 (10) | 0.0004 (10) |
| C13 | 0.0338 (13) | 0.0312 (14) | 0.0356 (15) | 0.0037 (10) | 0.0067 (11) | −0.0026 (10) |
| N1 | 0.0341 (11) | 0.0433 (13) | 0.0357 (12) | −0.0029 (9) | 0.0086 (9) | −0.0001 (9) |
| N2 | 0.0437 (12) | 0.0526 (14) | 0.0371 (13) | 0.0034 (10) | 0.0126 (10) | −0.0035 (10) |
| N3 | 0.0321 (10) | 0.0442 (12) | 0.0288 (11) | 0.0016 (9) | 0.0003 (9) | 0.0042 (9) |
| C5 | 0.0393 (15) | 0.0540 (18) | 0.0499 (18) | −0.0090 (12) | 0.0040 (12) | 0.0069 (14) |
| N5 | 0.0414 (13) | 0.0517 (14) | 0.0338 (13) | 0.0032 (10) | 0.0004 (10) | −0.0005 (10) |
| N6 | 0.0327 (11) | 0.0435 (13) | 0.0438 (14) | −0.0038 (9) | 0.0023 (10) | 0.0059 (10) |
| O1 | 0.0362 (9) | 0.0445 (10) | 0.0308 (10) | −0.0009 (7) | −0.0026 (7) | −0.0054 (7) |
| O2 | 0.0399 (10) | 0.0552 (11) | 0.0313 (10) | −0.0055 (8) | 0.0056 (7) | −0.0066 (8) |
| O3 | 0.0325 (9) | 0.0561 (11) | 0.0400 (10) | −0.0091 (8) | 0.0079 (7) | −0.0084 (8) |
| O4 | 0.0395 (10) | 0.0890 (14) | 0.0408 (11) | −0.0047 (9) | −0.0063 (8) | −0.0048 (9) |
| O5 | 0.0499 (11) | 0.1146 (17) | 0.0361 (11) | −0.0057 (10) | 0.0133 (9) | −0.0036 (10) |
| O6 | 0.0351 (10) | 0.1012 (16) | 0.0635 (13) | −0.0075 (10) | 0.0156 (9) | −0.0121 (11) |
| O7 | 0.0430 (10) | 0.0799 (14) | 0.0453 (11) | −0.0182 (9) | −0.0029 (8) | −0.0108 (10) |
| Zn1 | 0.0355 (2) | 0.0455 (3) | 0.0352 (3) | 0 | 0.00566 (17) | 0 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—H1 | 0.93 | C8—H8 | 0.93 |
| C1—C2 | 1.331 (4) | C8—C9 | 1.367 (3) |
| C1—N1 | 1.377 (3) | C9—C10 | 1.435 (3) |
| C2—H2 | 0.93 | C9—N6 | 1.462 (3) |
| C2—N2 | 1.362 (3) | C10—C11 | 1.440 (3) |
| C3—H3 | 0.93 | C10—O1 | 1.280 (3) |
| C3—N1 | 1.320 (3) | C11—C12 | 1.375 (3) |
| C3—N2 | 1.328 (3) | C11—C13 | 1.480 (3) |
| C4—H4 | 0.93 | C12—H12 | 0.93 |
| C4—N4 | 1.322 (3) | C13—O2 | 1.319 (3) |
| C4—N3 | 1.319 (3) | C13—O3 | 1.220 (3) |
| N4—C5 | 1.361 (3) | N2—H2a | 0.967 (2) |
| N4—H4a | 0.9427 (18) | C5—H5 | 0.93 |
| C6—H6 | 0.93 | N5—O4 | 1.231 (3) |
| C6—N3 | 1.381 (3) | N5—O5 | 1.223 (3) |
| C6—C5 | 1.335 (3) | N6—O6 | 1.231 (3) |
| C7—C8 | 1.378 (3) | N6—O7 | 1.217 (3) |
| C7—C12 | 1.377 (3) | O1—H1a | 1.4955 (15) |
| C7—N5 | 1.454 (3) | O2—H1a | 1.0386 (15) |
| | | | |
| H1—C1—C2 | 124.94 | C9—C10—O1 | 125.18 (18) |
| H1—C1—N1 | 124.94 | C11—C10—O1 | 120.2 (2) |
| C2—C1—N1 | 110.1 (2) | C10—C11—C12 | 121.4 (2) |
| C1—C2—H2 | 126.81 | C10—C11—C13 | 120.78 (19) |
| C1—C2—N2 | 106.4 (2) | C12—C11—C13 | 117.85 (18) |
| H2—C2—N2 | 126.81 | C7—C12—C11 | 120.69 (19) |
| H3—C3—N1 | 124.28 | C7—C12—H12 | 119.65 |
| H3—C3—N2 | 124.28 | C11—C12—H12 | 119.66 |
| N1—C3—N2 | 111.44 (19) | C11—C13—O2 | 117.03 (18) |
| H4—C4—N4 | 124.35 | C11—C13—O3 | 122.6 (2) |
| H4—C4—N3 | 124.35 | O2—C13—O3 | 120.3 (2) |
| N4—C4—N3 | 111.3 (2) | C1—N1—C3 | 104.66 (19) |
| C4—N4—C5 | 107.74 (19) | C2—N2—C3 | 107.4 (2) |
| C4—N4—H4a | 133.8 (2) | C2—N2—H2a | 121.3 (2) |
| C5—N4—H4a | 118.5 (2) | C3—N2—H2a | 131.3 (2) |
| H6—C6—N3 | 125.29 | C4—N3—C6 | 105.04 (18) |
| H6—C6—C5 | 125.29 | N4—C5—C6 | 106.5 (2) |
| N3—C6—C5 | 109.4 (2) | N4—C5—H5 | 126.75 |
| C8—C7—C12 | 120.8 (2) | C6—C5—H5 | 126.75 |
| C8—C7—N5 | 119.8 (2) | C7—N5—O4 | 117.82 (19) |
| C12—C7—N5 | 119.40 (19) | C7—N5—O5 | 119.10 (18) |
| C7—C8—H8 | 120.27 | O4—N5—O5 | 123.08 (19) |
| C7—C8—C9 | 119.5 (2) | C9—N6—O6 | 117.66 (19) |
| H8—C8—C9 | 120.27 | C9—N6—O7 | 119.73 (19) |
| C8—C9—C10 | 123.06 (18) | O6—N6—O7 | 122.61 (18) |
| C8—C9—N6 | 116.8 (2) | C10—O1—H1a | 98.67 (13) |

| | | | |
|---|---|---|---|
| C10—C9—N6 | 120.16 (19) | C13—O2—H1a | 102.66 (16) |
| C9—C10—C11 | 114.56 (19) | O1—H1a—O2 | 160.37 (10) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···O3$^{i}$ | 0.93 | 2.47 | 3.327 (3) | 154 |
| O2—H1*a*···C10 | 1.0386 (15) | 2.110 (2) | 2.820 (3) | 123.5 (1) |
| O2—H1*a*···O1 | 1.0386 (15) | 1.4955 (15) | 2.498 (2) | 160.4 (1) |
| N2—H2*a*···O3 | 0.967 (2) | 1.8902 (16) | 2.838 (3) | 165.87 (12) |
| N4—H4*a*···O1$^{ii}$ | 0.9427 (18) | 1.9236 (14) | 2.784 (2) | 150.60 (13) |
| N4—H4*a*···O7$^{ii}$ | 0.9427 (18) | 2.4336 (18) | 2.873 (3) | 108.36 (13) |

Symmetry codes: (i) *x*, −*y*+1, *z*+1/2; (ii) −*x*+3/2, *y*+1/2, −*z*+3/2.

### 3,5-Dinitrosalicylic acid–phenazine (1/1) (LUDFUL)

*Crystal data*

$C_7H_4N_2O_7·C_{12}H_8N_2$
$M_r$ = 408.33
Monoclinic, $P2_1/a$
*a* = 14.8002 (15) Å
*b* = 7.4029 (16) Å
*c* = 16.0091 (16) Å
*β* = 96.395 (8)°
*V* = 1743.1 (5) Å$^3$
*Z* = 4
*F*(000) = 840
$D_x$ = 1.556 Mg m$^{-3}$
Melting point: 471 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 25 reflections
*θ* = 5–12°
*μ* = 0.12 mm$^{-1}$
*T* = 293 K
Rhombic, yellow
0.36 × 0.34 × 0.26 mm

*Data collection*

Enraf-Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
w scans
8396 measured reflections
4202 independent reflections
1587 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.056
$\theta_{max}$ = 28.0°, $\theta_{min}$ = 1.5°
*h* = 0→19
*k* = −9→9
*l* = −21→21
3 standard reflections every 150 reflections
intensity decay: 2%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)]$ = 0.044
$wR(F)$ = 0.083
*S* = 1.08
4202 reflections
274 parameters
0 restraints
40 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$
Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 5100 (500)

*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**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. Number of fixed parameters: 6

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O3 | 0.98761 (10) | 0.2874 (2) | 0.46747 (9) | 0.0599 (6) |
| O1 | 0.85079 (8) | 0.4309 (2) | 0.24121 (9) | 0.0539 (6) |
| C1 | 0.98636 (12) | 0.4161 (3) | 0.33159 (12) | 0.0373 (7) |
| O7 | 1.12739 (10) | 0.6537 (3) | 0.15665 (10) | 0.0716 (7) |
| C4 | 1.17200 (13) | 0.4785 (3) | 0.36328 (13) | 0.0421 (8) |
| H4a | 1.234071 | 0.501018 | 0.373432 | 0.0506* |
| C5 | 1.12429 (13) | 0.5256 (3) | 0.28746 (12) | 0.0378 (7) |
| O2 | 0.84456 (9) | 0.3110 (2) | 0.36758 (9) | 0.0632 (7) |
| C2 | 1.03254 (14) | 0.3661 (3) | 0.40995 (13) | 0.0430 (8) |
| C3 | 1.12654 (13) | 0.3980 (3) | 0.42348 (12) | 0.0423 (8) |
| N1 | 1.18089 (15) | 0.3455 (3) | 0.50174 (12) | 0.0665 (9) |
| C7 | 0.88704 (14) | 0.3818 (3) | 0.31414 (14) | 0.0457 (9) |
| C6 | 1.03232 (13) | 0.4947 (3) | 0.27113 (12) | 0.0386 (7) |
| H6a | 1.001506 | 0.526882 | 0.219465 | 0.0463* |
| O6 | 1.25212 (10) | 0.6448 (3) | 0.23814 (10) | 0.0804 (8) |
| O4 | 1.14445 (14) | 0.2742 (3) | 0.55587 (13) | 0.1291 (12) |
| N2 | 1.17129 (12) | 0.6128 (3) | 0.22290 (12) | 0.0504 (8) |
| O5 | 1.26041 (12) | 0.3839 (3) | 0.51002 (10) | 0.0996 (10) |
| N3 | 0.68389 (10) | 0.3729 (2) | 0.18982 (10) | 0.0388 (6) |
| N4 | 0.50803 (11) | 0.3575 (3) | 0.10509 (11) | 0.0517 (7) |
| C17 | 0.57960 (14) | 0.2988 (3) | 0.06927 (13) | 0.0505 (9) |
| C19 | 0.61248 (13) | 0.4326 (3) | 0.22726 (12) | 0.0377 (7) |
| C8 | 0.74378 (15) | 0.2489 (3) | 0.06912 (15) | 0.0552 (9) |
| H8a | 0.802687 | 0.252957 | 0.096215 | 0.0663* |
| C16 | 0.66987 (13) | 0.3064 (3) | 0.11118 (13) | 0.0405 (8) |
| C18 | 0.52369 (13) | 0.4267 (3) | 0.18296 (13) | 0.0413 (8) |
| C12 | 0.45074 (14) | 0.4976 (3) | 0.22251 (15) | 0.0533 (9) |
| H12a | 0.392387 | 0.498106 | 0.194132 | 0.064* |
| C14 | 0.55304 (15) | 0.5655 (3) | 0.34545 (14) | 0.0522 (9) |
| H14a | 0.561295 | 0.610201 | 0.400052 | 0.0627* |
| C13 | 0.46523 (15) | 0.5645 (3) | 0.30108 (15) | 0.0555 (10) |
| H13a | 0.416606 | 0.610633 | 0.326453 | 0.0666* |
| C15 | 0.62592 (14) | 0.5020 (3) | 0.30951 (13) | 0.0442 (8) |
| H15a | 0.683759 | 0.504497 | 0.338918 | 0.053* |

| | | | | |
|---|---|---|---|---|
| C10 | 0.6400 (2) | 0.1763 (4) | −0.05197 (16) | 0.0789 (12) |
| H10a | 0.631177 | 0.131533 | −0.10654 | 0.0946* |
| C11 | 0.56781 (17) | 0.2291 (3) | −0.01395 (15) | 0.0689 (11) |
| H11a | 0.509671 | 0.219906 | −0.042373 | 0.0827* |
| C9 | 0.72833 (18) | 0.1879 (3) | −0.01060 (16) | 0.0685 (12) |
| H9a | 0.777245 | 0.152877 | −0.038619 | 0.0822* |
| H3a | 0.919191 | 0.279002 | 0.440153 | 0.138 (12)* |
| H1a | 0.773139 | 0.402666 | 0.229166 | 0.105 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O3 | 0.0634 (10) | 0.0734 (13) | 0.0442 (9) | −0.0001 (9) | 0.0122 (8) | 0.0094 (9) |
| O1 | 0.0315 (8) | 0.0749 (13) | 0.0545 (10) | −0.0027 (8) | 0.0009 (7) | 0.0096 (9) |
| C1 | 0.0315 (11) | 0.0413 (14) | 0.0394 (12) | 0.0004 (10) | 0.0046 (10) | −0.0033 (11) |
| O7 | 0.0556 (10) | 0.1066 (17) | 0.0520 (10) | −0.0111 (10) | 0.0040 (8) | 0.0199 (11) |
| C4 | 0.0326 (11) | 0.0468 (15) | 0.0457 (13) | 0.0043 (11) | −0.0016 (11) | −0.0111 (12) |
| C5 | 0.0325 (11) | 0.0414 (15) | 0.0398 (12) | 0.0028 (10) | 0.0059 (10) | −0.0035 (11) |
| O2 | 0.0445 (9) | 0.0836 (14) | 0.0637 (10) | −0.0069 (9) | 0.0161 (8) | 0.0161 (10) |
| C2 | 0.0493 (13) | 0.0426 (15) | 0.0377 (12) | 0.0029 (12) | 0.0073 (11) | −0.0033 (12) |
| C3 | 0.0449 (12) | 0.0468 (16) | 0.0337 (12) | 0.0128 (11) | −0.0024 (10) | −0.0056 (12) |
| N1 | 0.0646 (15) | 0.090 (2) | 0.0420 (13) | 0.0141 (14) | −0.0078 (12) | −0.0014 (13) |
| C7 | 0.0413 (13) | 0.0461 (16) | 0.0509 (14) | −0.0004 (12) | 0.0098 (11) | −0.0019 (13) |
| C6 | 0.0334 (11) | 0.0433 (14) | 0.0381 (12) | 0.0051 (10) | −0.0003 (10) | −0.0035 (11) |
| O6 | 0.0328 (8) | 0.1225 (17) | 0.0871 (12) | −0.0130 (10) | 0.0128 (8) | 0.0119 (12) |
| O4 | 0.1041 (16) | 0.197 (3) | 0.0807 (15) | −0.0108 (16) | −0.0151 (13) | 0.0758 (17) |
| N2 | 0.0369 (11) | 0.0590 (15) | 0.0567 (13) | −0.0011 (11) | 0.0116 (10) | −0.0046 (12) |
| O5 | 0.0575 (11) | 0.177 (2) | 0.0581 (11) | 0.0108 (14) | −0.0193 (9) | −0.0056 (13) |
| N3 | 0.0310 (9) | 0.0431 (12) | 0.0419 (10) | −0.0041 (9) | 0.0024 (8) | 0.0009 (10) |
| N4 | 0.0446 (11) | 0.0533 (14) | 0.0545 (12) | 0.0017 (10) | −0.0065 (9) | −0.0035 (11) |
| C17 | 0.0538 (14) | 0.0491 (17) | 0.0469 (14) | 0.0046 (13) | −0.0024 (12) | −0.0024 (13) |
| C19 | 0.0354 (12) | 0.0362 (14) | 0.0412 (13) | −0.0040 (11) | 0.0029 (10) | 0.0023 (11) |
| C8 | 0.0564 (15) | 0.0521 (18) | 0.0588 (16) | 0.0030 (13) | 0.0136 (13) | −0.0013 (13) |
| C16 | 0.0459 (13) | 0.0339 (14) | 0.0419 (13) | −0.0006 (11) | 0.0057 (11) | 0.0018 (11) |
| C18 | 0.0352 (11) | 0.0378 (15) | 0.0504 (13) | −0.0009 (11) | 0.0032 (10) | −0.0008 (12) |
| C12 | 0.0355 (13) | 0.0510 (17) | 0.0730 (17) | 0.0020 (12) | 0.0041 (12) | −0.0052 (14) |
| C14 | 0.0630 (15) | 0.0471 (17) | 0.0484 (14) | −0.0037 (14) | 0.0143 (13) | −0.0049 (13) |
| C13 | 0.0457 (14) | 0.0512 (18) | 0.0728 (18) | 0.0032 (13) | 0.0208 (13) | −0.0040 (15) |
| C15 | 0.0419 (13) | 0.0481 (16) | 0.0418 (13) | −0.0029 (12) | 0.0012 (11) | 0.0047 (12) |
| C10 | 0.107 (2) | 0.078 (2) | 0.0504 (16) | 0.009 (2) | 0.0051 (17) | −0.0198 (16) |
| C11 | 0.0775 (19) | 0.071 (2) | 0.0533 (17) | 0.0057 (16) | −0.0147 (14) | −0.0148 (15) |
| C9 | 0.089 (2) | 0.060 (2) | 0.0602 (18) | 0.0060 (17) | 0.0270 (15) | −0.0069 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O3—C2 | 1.329 (3) | N4—C17 | 1.332 (3) |
| O3—H3a | 1.0592 (14) | N4—C18 | 1.344 (3) |
| O1—C7 | 1.282 (3) | C17—C16 | 1.428 (3) |

| | | | |
|---|---|---|---|
| O1—H1a | 1.1628 (13) | C17—C11 | 1.421 (3) |
| C1—C2 | 1.410 (3) | C19—C18 | 1.423 (3) |
| C1—C7 | 1.487 (3) | C19—C15 | 1.407 (3) |
| C1—C6 | 1.373 (3) | C8—H8a | 0.93 |
| O7—N2 | 1.219 (2) | C8—C16 | 1.413 (3) |
| C4—H4a | 0.93 | C8—C9 | 1.349 (3) |
| C4—C5 | 1.379 (3) | C18—C12 | 1.412 (3) |
| C4—C3 | 1.371 (3) | C12—H12a | 0.93 |
| C5—C6 | 1.376 (3) | C12—C13 | 1.347 (3) |
| C5—N2 | 1.459 (3) | C14—H14a | 0.93 |
| O2—C7 | 1.234 (3) | C14—C13 | 1.410 (3) |
| C2—C3 | 1.404 (3) | C14—C15 | 1.361 (3) |
| C3—N1 | 1.464 (3) | C13—H13a | 0.93 |
| N1—O4 | 1.194 (3) | C15—H15a | 0.93 |
| N1—O5 | 1.204 (3) | C10—H10a | 0.93 |
| C6—H6a | 0.93 | C10—C11 | 1.346 (4) |
| O6—N2 | 1.217 (2) | C10—C9 | 1.400 (4) |
| N3—C19 | 1.346 (3) | C11—H11a | 0.93 |
| N3—C16 | 1.346 (3) | C9—H9a | 0.93 |

| | | | |
|---|---|---|---|
| C2—O3—H3a | 105.60 (13) | C16—C17—C11 | 117.8 (2) |
| C7—O1—H1a | 113.94 (15) | N3—C19—C18 | 119.68 (18) |
| C2—C1—C7 | 119.58 (18) | N3—C19—C15 | 120.03 (17) |
| C2—C1—C6 | 120.63 (17) | C18—C19—C15 | 120.28 (19) |
| C7—C1—C6 | 119.79 (17) | H8a—C8—C16 | 120.23 |
| H4a—C4—C5 | 120.49 | H8a—C8—C9 | 120.23 |
| H4a—C4—C3 | 120.49 | C16—C8—C9 | 119.5 (2) |
| C5—C4—C3 | 119.01 (18) | N3—C16—C17 | 119.52 (18) |
| C4—C5—C6 | 121.58 (19) | N3—C16—C8 | 120.61 (17) |
| C4—C5—N2 | 119.83 (17) | C17—C16—C8 | 119.86 (19) |
| C6—C5—N2 | 118.58 (17) | N4—C18—C19 | 121.90 (19) |
| C7—O2—H3a | 102.24 (13) | N4—C18—C12 | 119.74 (18) |
| O3—C2—C1 | 120.09 (17) | C19—C18—C12 | 118.35 (19) |
| O3—C2—C3 | 122.05 (17) | C18—C12—H12a | 119.82 |
| C1—C2—C3 | 117.84 (19) | C18—C12—C13 | 120.35 (19) |
| C4—C3—C2 | 121.34 (18) | H12a—C12—C13 | 119.82 |
| C4—C3—N1 | 116.79 (18) | H14a—C14—C13 | 119.52 |
| C2—C3—N1 | 121.86 (19) | H14a—C14—C15 | 119.52 |
| C3—N1—O4 | 119.3 (2) | C13—C14—C15 | 121.0 (2) |
| C3—N1—O5 | 118.0 (2) | C12—C13—C14 | 121.0 (2) |
| O4—N1—O5 | 122.7 (2) | C12—C13—H13a | 119.52 |
| O1—C7—C1 | 115.30 (19) | C14—C13—H13a | 119.52 |
| O1—C7—O2 | 123.93 (18) | C19—C15—C14 | 119.05 (18) |
| C1—C7—O2 | 120.76 (18) | C19—C15—H15a | 120.48 |
| C1—C6—C5 | 119.59 (17) | C14—C15—H15a | 120.48 |
| C1—C6—H6a | 120.21 | H10a—C10—C11 | 119.51 |
| C5—C6—H6a | 120.2 | H10a—C10—C9 | 119.51 |
| O7—N2—C5 | 118.45 (16) | C11—C10—C9 | 121.0 (2) |

| | | | |
|---|---|---|---|
| O7—N2—O6 | 122.87 (19) | C17—C11—C10 | 120.6 (2) |
| C5—N2—O6 | 118.66 (17) | C17—C11—H11a | 119.71 |
| C19—N3—C16 | 119.31 (15) | C10—C11—H11a | 119.71 |
| C19—N3—H1a | 119.38 (14) | C8—C9—C10 | 121.3 (3) |
| C16—N3—H1a | 120.79 (14) | C8—C9—H9a | 119.37 |
| C17—N4—C18 | 117.41 (17) | C10—C9—H9a | 119.37 |
| N4—C17—C16 | 122.14 (19) | O3—H3a—O2 | 151.72 (10) |
| N4—C17—C11 | 120.09 (19) | O1—H1a—N3 | 163.24 (10) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C13—H13*a*···O4$^{i}$ | 0.93 | 2.49 | 3.334 (3) | 151 |
| O3—H3*a*···O2 | 1.0592 (14) | 1.5297 (14) | 2.5132 (19) | 151.72 (10) |
| O1—H1*a*···N3 | 1.1628 (13) | 1.4160 (14) | 2.5515 (19) | 163.24 (10) |

Symmetry code: (i) −*x*+3/2, *y*+1/2, −*z*+1.

## 2-Amino-5-methylpyridinium 2-hydroxy-3,5-dinitrobenzoate (NUQVEB)

*Crystal data*

$C_6H_9N_2^+·C_7H_3N_2O_7^-$
$M_r$ = 336.27
Triclinic, $P\overline{1}$
Hall symbol: -P 1
*a* = 5.8673 (7) Å
*b* = 8.0991 (9) Å
*c* = 15.2437 (17) Å
*α* = 86.844 (3)°
*β* = 84.252 (3)°
*γ* = 81.209 (3)°
*V* = 711.69 (14) Å$^3$
*Z* = 2
*F*(000) = 348
$D_x$ = 1.569 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5139 reflections
*θ* = 2.7–32.4°
*μ* = 0.13 mm$^{-1}$
*T* = 100 K
Block, yellow
0.29 × 0.14 × 0.08 mm

*Data collection*

Bruker APEX DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*φ* and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
$T_{min}$ = 0.963, $T_{max}$ = 0.990
12709 measured reflections
4943 independent reflections
3677 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 32.5°, $\theta_{min}$ = 1.3°
*h* = −8→8
*k* = −12→11
*l* = −22→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)]$ = 0.042
$wR(F)$ = 0.109
*S* = 2.06
4943 reflections
222 parameters
0 restraints
34 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$

$(\Delta/\sigma)_{max}$ = 0.009
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. Number of fixed parameters: 15

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.50355 (15) | 0.28624 (11) | 0.24318 (6) | 0.0153 (3) | |
| N2 | 0.82331 (16) | 0.41649 (12) | 0.19964 (6) | 0.0182 (3) | |
| C1 | 0.67739 (18) | 0.36376 (13) | 0.26429 (7) | 0.0152 (3) | |
| C2 | 0.35124 (18) | 0.22197 (14) | 0.30471 (7) | 0.0165 (3) | |
| H2 | 0.233313 | 0.167725 | 0.285731 | 0.0198* | |
| C3 | 0.36539 (18) | 0.23424 (14) | 0.39257 (7) | 0.0180 (3) | |
| C4 | 0.54266 (19) | 0.31918 (15) | 0.41642 (7) | 0.0200 (3) | |
| H4 | 0.556083 | 0.332079 | 0.477169 | 0.024* | |
| C5 | 0.69461 (19) | 0.38301 (14) | 0.35491 (7) | 0.0186 (3) | |
| H5 | 0.811136 | 0.439959 | 0.372833 | 0.0223* | |
| C6 | 0.2014 (2) | 0.16274 (17) | 0.46143 (8) | 0.0262 (4) | |
| H6a | 0.118019 | 0.252439 | 0.498084 | 0.0394* | |
| H6b | 0.090144 | 0.110743 | 0.432458 | 0.0394* | |
| H6c | 0.289083 | 0.078457 | 0.498608 | 0.0394* | |
| O1 | 0.17284 (13) | 0.61818 (10) | 0.14277 (5) | 0.0201 (2) | |
| O2 | 0.10549 (15) | 0.60964 (11) | 0.31689 (5) | 0.0252 (3) | |
| O3 | 0.28472 (15) | 0.76461 (12) | 0.38855 (5) | 0.0275 (3) | |
| O4 | 0.93312 (14) | 0.99538 (11) | 0.26216 (6) | 0.0242 (3) | |
| O5 | 0.99966 (15) | 1.02486 (11) | 0.11988 (6) | 0.0279 (3) | |
| O6 | 0.55543 (14) | 0.76370 (11) | −0.07523 (5) | 0.0224 (3) | |
| O7 | 0.26607 (14) | 0.63258 (10) | −0.01549 (5) | 0.0199 (2) | |
| N3 | 0.25052 (15) | 0.70323 (12) | 0.31961 (6) | 0.0172 (3) | |
| N4 | 0.89095 (16) | 0.97282 (12) | 0.18597 (6) | 0.0190 (3) | |
| C7 | 0.33912 (17) | 0.70052 (13) | 0.15501 (7) | 0.0141 (3) | |
| C8 | 0.38900 (17) | 0.74506 (13) | 0.23906 (7) | 0.0144 (3) | |
| C9 | 0.56997 (17) | 0.83259 (13) | 0.24874 (7) | 0.0156 (3) | |
| H9 | 0.601439 | 0.859704 | 0.305724 | 0.0187* | |
| C10 | 0.70326 (17) | 0.87968 (13) | 0.17493 (7) | 0.0154 (3) | |
| C11 | 0.66121 (18) | 0.84357 (13) | 0.09021 (7) | 0.0157 (3) | |
| H11 | 0.753695 | 0.879143 | 0.040052 | 0.0188* | |

| | | | | | |
|---|---|---|---|---|---|
| C12 | 0.48279 (17) | 0.75522 (13) | 0.08058 (7) | 0.0139 (3) | |
| C13 | 0.43623 (18) | 0.71649 (13) | −0.01006 (7) | 0.0165 (3) | |
| H1o7 | 0.20768 | 0.615457 | 0.041923 | 0.044 (6)* | 0.62 (3) |
| H1o1 | 0.186813 | 0.613081 | 0.081569 | 0.044 (6)* | 0.38 (3) |
| H2a | 0.809476 | 0.397973 | 0.143402 | 0.035 (4)* | |
| H2b | 0.928572 | 0.469891 | 0.211657 | 0.048 (5)* | |
| H1 | 0.481117 | 0.276133 | 0.186443 | 0.032 (4)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0166 (4) | 0.0168 (5) | 0.0130 (4) | −0.0032 (3) | −0.0023 (3) | −0.0006 (3) |
| N2 | 0.0194 (4) | 0.0221 (5) | 0.0147 (4) | −0.0085 (4) | −0.0004 (3) | −0.0009 (4) |
| C1 | 0.0166 (5) | 0.0137 (5) | 0.0152 (5) | −0.0012 (4) | −0.0022 (4) | −0.0004 (4) |
| C2 | 0.0142 (5) | 0.0162 (5) | 0.0192 (5) | −0.0026 (4) | −0.0015 (4) | −0.0007 (4) |
| C3 | 0.0182 (5) | 0.0180 (5) | 0.0168 (5) | −0.0011 (4) | 0.0004 (4) | 0.0009 (4) |
| C4 | 0.0239 (5) | 0.0239 (6) | 0.0127 (5) | −0.0043 (5) | −0.0035 (4) | −0.0007 (4) |
| C5 | 0.0201 (5) | 0.0201 (6) | 0.0169 (5) | −0.0054 (4) | −0.0041 (4) | −0.0004 (4) |
| C6 | 0.0238 (6) | 0.0324 (7) | 0.0219 (6) | −0.0077 (5) | 0.0028 (4) | 0.0047 (5) |
| O1 | 0.0192 (4) | 0.0259 (4) | 0.0176 (4) | −0.0108 (3) | −0.0027 (3) | −0.0010 (3) |
| O2 | 0.0275 (4) | 0.0281 (5) | 0.0220 (4) | −0.0141 (4) | 0.0034 (3) | −0.0013 (3) |
| O3 | 0.0290 (5) | 0.0434 (6) | 0.0120 (4) | −0.0097 (4) | −0.0019 (3) | −0.0062 (4) |
| O4 | 0.0215 (4) | 0.0234 (4) | 0.0302 (5) | −0.0036 (3) | −0.0110 (3) | −0.0066 (4) |
| O5 | 0.0227 (4) | 0.0276 (5) | 0.0354 (5) | −0.0122 (4) | −0.0009 (3) | 0.0022 (4) |
| O6 | 0.0280 (4) | 0.0278 (5) | 0.0128 (4) | −0.0102 (4) | 0.0001 (3) | −0.0006 (3) |
| O7 | 0.0229 (4) | 0.0252 (4) | 0.0139 (4) | −0.0096 (3) | −0.0034 (3) | −0.0010 (3) |
| N3 | 0.0168 (4) | 0.0200 (5) | 0.0145 (4) | −0.0020 (4) | −0.0017 (3) | −0.0002 (4) |
| N4 | 0.0154 (4) | 0.0150 (5) | 0.0275 (5) | −0.0027 (4) | −0.0051 (4) | −0.0025 (4) |
| C7 | 0.0135 (4) | 0.0136 (5) | 0.0150 (5) | −0.0010 (4) | −0.0022 (4) | −0.0010 (4) |
| C8 | 0.0145 (5) | 0.0155 (5) | 0.0128 (5) | −0.0016 (4) | 0.0000 (4) | −0.0003 (4) |
| C9 | 0.0144 (5) | 0.0156 (5) | 0.0169 (5) | 0.0000 (4) | −0.0038 (4) | −0.0031 (4) |
| C10 | 0.0128 (4) | 0.0131 (5) | 0.0210 (5) | −0.0030 (4) | −0.0035 (4) | −0.0018 (4) |
| C11 | 0.0148 (5) | 0.0137 (5) | 0.0179 (5) | −0.0009 (4) | −0.0007 (4) | −0.0005 (4) |
| C12 | 0.0145 (4) | 0.0141 (5) | 0.0132 (5) | −0.0020 (4) | −0.0018 (4) | −0.0009 (4) |
| C13 | 0.0190 (5) | 0.0153 (5) | 0.0151 (5) | −0.0021 (4) | −0.0026 (4) | −0.0012 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.3498 (15) | O1—H1o1 | 0.9310 (8) |
| N1—C2 | 1.3674 (14) | O2—N3 | 1.2280 (14) |
| N1—H1 | 0.8977 (9) | O3—N3 | 1.2338 (13) |
| N2—C1 | 1.3353 (14) | O4—N4 | 1.2402 (14) |
| N2—H2a | 0.8921 (9) | O5—N4 | 1.2273 (13) |
| N2—H2b | 0.8456 (10) | O6—C13 | 1.2340 (13) |
| C1—C5 | 1.4139 (15) | O7—C13 | 1.3022 (15) |
| C2—H2 | 0.95 | O7—H1o7 | 0.9185 (8) |
| C2—C3 | 1.3602 (16) | N3—C8 | 1.4564 (13) |
| C3—C4 | 1.4174 (17) | N4—C10 | 1.4544 (15) |

| | | | |
|---|---|---|---|
| C3—C6 | 1.5049 (16) | C7—C8 | 1.4197 (15) |
| C4—H4 | 0.95 | C7—C12 | 1.4357 (14) |
| C4—C5 | 1.3643 (16) | C8—C9 | 1.3874 (16) |
| C5—H5 | 0.95 | C9—H9 | 0.95 |
| C6—H6a | 0.98 | C9—C10 | 1.3750 (15) |
| C6—H6b | 0.98 | C10—C11 | 1.3934 (16) |
| C6—H6c | 0.98 | C11—H11 | 0.95 |
| H6a—H6b | 1.6003 | C11—C12 | 1.3787 (16) |
| H6a—H6c | 1.6003 | C12—C13 | 1.4939 (15) |
| H6b—H6c | 1.6003 | H2a—H2b | 1.4990 (2) |
| O1—C7 | 1.2964 (14) | | |

| | | | |
|---|---|---|---|
| C1—N1—C2 | 123.29 (9) | O2—N3—O3 | 122.52 (9) |
| C1—N1—H1 | 120.41 (9) | O2—N3—C8 | 119.61 (9) |
| C2—N1—H1 | 116.29 (10) | O3—N3—C8 | 117.87 (10) |
| C1—N2—H2a | 120.74 (11) | O4—N4—O5 | 123.30 (10) |
| C1—N2—H2b | 120.05 (10) | O4—N4—C10 | 118.02 (9) |
| H2a—N2—H2b | 119.20 (10) | O5—N4—C10 | 118.69 (10) |
| N1—C1—N2 | 118.98 (10) | O1—C7—C8 | 124.03 (9) |
| N1—C1—C5 | 117.27 (9) | O1—C7—C12 | 119.84 (10) |
| N2—C1—C5 | 123.75 (11) | C8—C7—C12 | 116.12 (10) |
| N1—C2—H2 | 119.38 | N3—C8—C7 | 121.67 (10) |
| N1—C2—C3 | 121.25 (11) | N3—C8—C9 | 116.48 (9) |
| H2—C2—C3 | 119.38 | C7—C8—C9 | 121.84 (9) |
| C2—C3—C4 | 116.54 (10) | C8—C9—H9 | 120.37 |
| C2—C3—C6 | 122.15 (11) | C8—C9—C10 | 119.26 (10) |
| C4—C3—C6 | 121.31 (10) | H9—C9—C10 | 120.37 |
| C3—C4—H4 | 118.95 | N4—C10—C9 | 118.68 (10) |
| C3—C4—C5 | 122.10 (10) | N4—C10—C11 | 119.25 (9) |
| H4—C4—C5 | 118.95 | C9—C10—C11 | 122.05 (10) |
| C1—C5—C4 | 119.51 (11) | C10—C11—H11 | 120.64 |
| C1—C5—H5 | 120.24 | C10—C11—C12 | 118.72 (9) |
| C4—C5—H5 | 120.24 | H11—C11—C12 | 120.64 |
| C3—C6—H6a | 109.47 | C7—C12—C11 | 121.98 (10) |
| C3—C6—H6b | 109.47 | C7—C12—C13 | 119.00 (10) |
| C3—C6—H6c | 109.47 | C11—C12—C13 | 119.02 (9) |
| H6a—C6—H6b | 109.47 | O6—C13—O7 | 123.12 (10) |
| H6a—C6—H6c | 109.47 | O6—C13—C12 | 120.31 (10) |
| H6b—C6—H6c | 109.47 | O7—C13—C12 | 116.57 (9) |
| C7—O1—H1o7 | 98.33 (6) | O1—H1o7—O7 | 161.55 (6) |
| C7—O1—H1o1 | 101.65 (8) | O7—H1o7—H1o1 | 166.67 (6) |
| C13—O7—H1o7 | 104.71 (9) | O1—H1o1—O7 | 163.52 (6) |
| C13—O7—H1o1 | 99.43 (7) | O1—H1o1—H1o7 | 171.56 (5) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2···O4$^{i}$ | 0.95 | 2.47 | 3.4107 (16) | 169 |

| | | | | |
|---|---|---|---|---|
| C4—H4···O3$^{ii}$ | 0.95 | 2.38 | 3.2397 (15) | 151 |
| C5—H5···O2$^{iii}$ | 0.95 | 2.43 | 3.2361 (16) | 143 |
| O7—H1*o*7···O1 | 0.9185 (8) | 1.5313 (8) | 2.4202 (12) | 161.55 (6) |
| O1—H1*o*1···O7 | 0.9310 (8) | 1.5130 (8) | 2.4202 (12) | 163.52 (6) |
| N2—H2*a*···O7$^{iv}$ | 0.8921 (9) | 2.0783 (9) | 2.9655 (14) | 172.84 (6) |
| N2—H2*b*···O1$^{iii}$ | 0.8456 (10) | 2.1644 (9) | 2.8526 (14) | 138.40 (6) |
| N2—H2*b*···O2$^{iii}$ | 0.8456 (10) | 2.4133 (10) | 3.1741 (14) | 150.02 (6) |
| N1—H1···O6$^{iv}$ | 0.8977 (9) | 1.7828 (9) | 2.6781 (13) | 174.83 (6) |

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

## 3,5-Diamino-6-(2,3-dichlorophenyl)-1,2,4-triazin-2-ium 3,5-dinitro-2-hydroxybenzoate *N*,*N*-dimethylformamide monosolvate (QIQJAD)

*Crystal data*

$C_9H_8Cl_2N_5^+·C_7H_3N_2O_7^-·C_3H_7NO$
$M_r$ = 557.31
Triclinic, $P\overline{1}$
Hall symbol: -P 1
*a* = 10.0227 (5) Å
*b* = 10.5507 (5) Å
*c* = 12.5359 (6) Å
*α* = 81.858 (1)°
*β* = 71.888 (1)°
*γ* = 70.009 (1)°
*V* = 1183.1 (1) Å$^3$
*Z* = 2
*F*(000) = 572
$D_x$ = 1.564 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6413 reflections
*θ* = 2.3–28.2°
*μ* = 0.34 mm$^{-1}$
*T* = 294 K
Plate, colourless
0.16 × 0.14 × 0.08 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
$T_{min}$ = 0.93, $T_{max}$ = 0.97
13936 measured reflections
5507 independent reflections
4441 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.019
$\theta_{max}$ = 28.0°, $\theta_{min}$ = 1.7°
*h* = −12→13
*k* = −13→13
*l* = −16→16

*Refinement*

Refinement on $F^2$
$R[F > 3\sigma(F)]$ = 0.056
$wR(F)$ = 0.147
*S* = 3.41
5507 reflections
340 parameters
0 restraints
48 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.020
$\Delta\rho_{max}$ = 0.80 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$

*Special details*

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

**Refinement**. Number of fixed parameters: 18

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.32107 (18) | 1.06764 (19) | 0.15844 (16) | 0.0464 (7) |
| C2 | 0.2718 (2) | 1.0232 (2) | 0.08378 (15) | 0.0485 (7) |
| C3 | 0.2713 (2) | 1.0903 (2) | −0.02046 (16) | 0.0529 (8) |
| C4 | 0.3231 (2) | 1.2001 (2) | −0.04951 (19) | 0.0621 (9) |
| H4 | 0.323127 | 1.245173 | −0.11886 | 0.0745* |
| C5 | 0.3741 (2) | 1.2420 (2) | 0.02358 (19) | 0.0632 (10) |
| H5 | 0.410127 | 1.314857 | 0.001937 | 0.0758* |
| C6 | 0.3745 (2) | 1.1807 (2) | 0.12857 (17) | 0.0537 (8) |
| H6 | 0.40816 | 1.211924 | 0.177727 | 0.0645* |
| C7 | 0.32753 (18) | 0.99894 (19) | 0.26974 (15) | 0.0437 (7) |
| C8 | 0.35068 (19) | 0.8912 (2) | 0.47363 (15) | 0.0449 (7) |
| C9 | 0.19780 (18) | 0.99408 (18) | 0.36291 (15) | 0.0418 (7) |
| N1 | 0.45735 (16) | 0.95248 (17) | 0.28531 (13) | 0.0488 (6) |
| N2 | 0.46755 (16) | 0.89842 (17) | 0.38722 (13) | 0.0484 (6) |
| N3 | 0.37354 (18) | 0.8352 (2) | 0.56859 (14) | 0.0613 (8) |
| N4 | 0.21299 (15) | 0.93978 (16) | 0.46191 (12) | 0.0454 (6) |
| N5 | 0.06397 (16) | 1.04599 (17) | 0.35102 (13) | 0.0504 (7) |
| Cl1 | 0.21444 (7) | 0.88366 (6) | 0.11855 (5) | 0.0685 (3) |
| Cl2 | 0.20368 (7) | 1.04039 (8) | −0.11161 (5) | 0.0772 (3) |
| C10 | 0.7671 (2) | 0.7509 (2) | 0.50260 (17) | 0.0495 (8) |
| C11 | 0.92521 (19) | 0.68875 (18) | 0.50532 (16) | 0.0456 (8) |
| C12 | 1.0399 (2) | 0.68056 (19) | 0.40501 (17) | 0.0482 (8) |
| C13 | 1.1855 (2) | 0.6217 (2) | 0.4120 (2) | 0.0565 (9) |
| C14 | 1.2169 (3) | 0.5728 (2) | 0.5120 (2) | 0.0650 (11) |
| H14 | 1.31429 | 0.535992 | 0.515021 | 0.078* |
| C15 | 1.1014 (3) | 0.5795 (2) | 0.6070 (2) | 0.0608 (10) |
| C16 | 0.9560 (2) | 0.63637 (19) | 0.60559 (18) | 0.0537 (9) |
| H16 | 0.879592 | 0.639442 | 0.671313 | 0.0645* |
| N6 | 1.3082 (2) | 0.6096 (2) | 0.3083 (2) | 0.0763 (10) |
| N7 | 1.1324 (3) | 0.5213 (2) | 0.7146 (3) | 0.0870 (14) |
| O1 | 0.74835 (14) | 0.79734 (16) | 0.40757 (12) | 0.0638 (7) |
| O2 | 0.66684 (16) | 0.75284 (17) | 0.58991 (13) | 0.0695 (7) |
| O3 | 1.01381 (16) | 0.72881 (16) | 0.30779 (13) | 0.0651 (7) |
| O4 | 1.3027 (2) | 0.5624 (2) | 0.22939 (18) | 0.0962 (10) |
| O5 | 1.4096 (2) | 0.6460 (3) | 0.3106 (2) | 0.1311 (14) |
| O6 | 1.2621 (3) | 0.4654 (3) | 0.7114 (2) | 0.1272 (14) |
| O7 | 1.0297 (3) | 0.5356 (3) | 0.7980 (2) | 0.1153 (15) |
| C17 | 0.8968 (2) | 0.2419 (2) | 0.13898 (19) | 0.0638 (10) |

| | | | | |
|---|---|---|---|---|
| H17 | 0.997049 | 0.225753 | 0.10284 | 0.0765* |
| C18 | 0.6486 (3) | 0.3564 (4) | 0.1366 (3) | 0.1002 (16) |
| H18a | 0.600566 | 0.362237 | 0.079827 | 0.1503* |
| H18b | 0.612084 | 0.441665 | 0.171497 | 0.1503* |
| H18c | 0.628154 | 0.287766 | 0.192178 | 0.1503* |
| C19 | 0.8613 (4) | 0.3905 (3) | −0.0204 (2) | 0.0959 (16) |
| H19a | 0.812684 | 0.38463 | −0.07367 | 0.1439* |
| H19b | 0.842404 | 0.483699 | −0.008394 | 0.1439* |
| H19c | 0.965894 | 0.347377 | −0.048929 | 0.1439* |
| N8 | 0.8043 (2) | 0.32305 (17) | 0.08649 (14) | 0.0588 (8) |
| O8 | 0.86513 (17) | 0.18476 (18) | 0.23111 (12) | 0.0693 (7) |
| H3n | 0.459992 | 0.808642 | 0.579383 | 0.067 (7)* |
| H4n | 0.300684 | 0.831649 | 0.62665 | 0.058 (6)* |
| H2n | 0.560448 | 0.867423 | 0.392878 | 0.069 (7)* |
| H5n | −0.01125 | 1.040769 | 0.406749 | 0.056 (6)* |
| H6n | 0.043449 | 1.086703 | 0.2906 | 0.052 (6)* |
| H3o | 0.911378 | 0.756483 | 0.330791 | 0.131 (12)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0300 (8) | 0.0541 (11) | 0.0483 (10) | −0.0081 (8) | −0.0060 (7) | −0.0055 (8) |
| C2 | 0.0354 (9) | 0.0578 (11) | 0.0464 (10) | −0.0118 (8) | −0.0044 (8) | −0.0068 (8) |
| C3 | 0.0364 (9) | 0.0682 (13) | 0.0443 (10) | −0.0090 (9) | −0.0049 (8) | −0.0047 (9) |
| C4 | 0.0457 (11) | 0.0737 (15) | 0.0546 (12) | −0.0053 (10) | −0.0119 (9) | −0.0038 (11) |
| C5 | 0.0580 (13) | 0.0599 (13) | 0.0701 (14) | −0.0224 (11) | −0.0153 (11) | 0.0054 (11) |
| C6 | 0.0442 (10) | 0.0589 (12) | 0.0502 (11) | −0.0107 (9) | −0.0112 (9) | 0.0032 (9) |
| C7 | 0.0323 (9) | 0.0527 (11) | 0.0433 (10) | −0.0115 (8) | −0.0076 (7) | −0.0053 (8) |
| C8 | 0.0316 (8) | 0.0564 (11) | 0.0446 (10) | −0.0113 (8) | −0.0096 (7) | −0.0050 (8) |
| C9 | 0.0305 (8) | 0.0502 (10) | 0.0430 (9) | −0.0112 (7) | −0.0074 (7) | −0.0072 (8) |
| N1 | 0.0326 (8) | 0.0622 (10) | 0.0469 (9) | −0.0126 (7) | −0.0079 (6) | −0.0007 (7) |
| N2 | 0.0282 (7) | 0.0679 (10) | 0.0450 (9) | −0.0113 (7) | −0.0098 (6) | −0.0005 (7) |
| N3 | 0.0343 (8) | 0.0949 (14) | 0.0468 (9) | −0.0152 (9) | −0.0110 (7) | 0.0078 (9) |
| N4 | 0.0293 (7) | 0.0618 (10) | 0.0417 (8) | −0.0119 (7) | −0.0077 (6) | −0.0027 (7) |
| N5 | 0.0307 (7) | 0.0730 (11) | 0.0421 (9) | −0.0106 (7) | −0.0102 (7) | −0.0001 (8) |
| Cl1 | 0.0784 (4) | 0.0786 (4) | 0.0600 (3) | −0.0419 (3) | −0.0150 (3) | −0.0059 (3) |
| Cl2 | 0.0721 (4) | 0.1162 (5) | 0.0522 (3) | −0.0373 (4) | −0.0190 (3) | −0.0090 (3) |
| C10 | 0.0386 (10) | 0.0504 (11) | 0.0597 (12) | −0.0073 (8) | −0.0180 (9) | −0.0107 (9) |
| C11 | 0.0404 (10) | 0.0417 (10) | 0.0597 (12) | −0.0103 (8) | −0.0219 (9) | −0.0067 (8) |
| C12 | 0.0395 (10) | 0.0464 (10) | 0.0641 (12) | −0.0136 (8) | −0.0218 (9) | −0.0030 (9) |
| C13 | 0.0384 (10) | 0.0512 (11) | 0.0826 (15) | −0.0101 (8) | −0.0220 (10) | −0.0101 (10) |
| C14 | 0.0504 (12) | 0.0533 (12) | 0.1065 (19) | −0.0093 (10) | −0.0465 (13) | −0.0135 (12) |
| C15 | 0.0692 (15) | 0.0506 (12) | 0.0797 (15) | −0.0144 (10) | −0.0492 (13) | −0.0036 (10) |
| C16 | 0.0578 (12) | 0.0501 (11) | 0.0625 (13) | −0.0162 (9) | −0.0287 (10) | −0.0069 (9) |
| N6 | 0.0372 (10) | 0.0779 (14) | 0.1062 (18) | −0.0082 (9) | −0.0156 (10) | −0.0157 (12) |
| N7 | 0.113 (2) | 0.0759 (15) | 0.1066 (19) | −0.0325 (14) | −0.0800 (17) | 0.0073 (14) |
| O1 | 0.0374 (7) | 0.0841 (11) | 0.0645 (9) | −0.0093 (7) | −0.0222 (7) | 0.0067 (8) |
| O2 | 0.0428 (8) | 0.0946 (12) | 0.0601 (9) | −0.0074 (8) | −0.0121 (7) | −0.0108 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O3 | 0.0427 (8) | 0.0802 (10) | 0.0653 (9) | −0.0151 (7) | −0.0148 (7) | 0.0074 (8) |
| O4 | 0.0575 (10) | 0.1277 (17) | 0.0882 (13) | −0.0110 (11) | −0.0135 (9) | −0.0195 (12) |
| O5 | 0.0520 (11) | 0.164 (2) | 0.181 (2) | −0.0480 (13) | 0.0026 (13) | −0.0623 (18) |
| O6 | 0.1283 (19) | 0.1273 (18) | 0.142 (2) | 0.0037 (15) | −0.1080 (17) | −0.0100 (15) |
| O7 | 0.142 (2) | 0.159 (2) | 0.0879 (15) | −0.0849 (19) | −0.0675 (16) | 0.0370 (15) |
| C17 | 0.0454 (11) | 0.0753 (15) | 0.0575 (13) | −0.0065 (10) | −0.0078 (10) | −0.0085 (11) |
| C18 | 0.0568 (15) | 0.129 (3) | 0.120 (2) | −0.0180 (16) | −0.0417 (16) | −0.009 (2) |
| C19 | 0.123 (3) | 0.0831 (19) | 0.0697 (17) | −0.0251 (18) | −0.0261 (16) | 0.0120 (14) |
| N8 | 0.0593 (11) | 0.0601 (11) | 0.0548 (10) | −0.0096 (9) | −0.0229 (9) | −0.0033 (8) |
| O8 | 0.0558 (9) | 0.0937 (12) | 0.0520 (9) | −0.0195 (8) | −0.0165 (7) | 0.0097 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.382 (3) | C13—C14 | 1.374 (4) |
| C1—C6 | 1.427 (3) | C13—N6 | 1.471 (3) |
| C1—C7 | 1.488 (3) | C14—H14 | 0.93 |
| C2—C3 | 1.396 (3) | C14—C15 | 1.369 (3) |
| C3—C4 | 1.385 (4) | C15—C16 | 1.378 (3) |
| C4—H4 | 0.93 | C15—N7 | 1.479 (4) |
| C4—C5 | 1.362 (4) | C16—H16 | 0.93 |
| C5—H5 | 0.93 | N6—O4 | 1.192 (4) |
| C5—C6 | 1.382 (3) | N6—O5 | 1.213 (4) |
| C6—H6 | 0.93 | N7—O6 | 1.221 (4) |
| C7—C9 | 1.464 (2) | N7—O7 | 1.202 (4) |
| C7—N1 | 1.291 (2) | O3—H3o | 0.9258 (14) |
| C8—N2 | 1.342 (2) | C17—H17 | 0.93 |
| C8—N3 | 1.303 (3) | C17—N8 | 1.305 (3) |
| C8—N4 | 1.345 (2) | C17—O8 | 1.226 (3) |
| C9—N4 | 1.322 (2) | C18—H18a | 0.96 |
| C9—N5 | 1.312 (2) | C18—H18b | 0.96 |
| N1—N2 | 1.343 (2) | C18—H18c | 0.96 |
| N2—H2n | 0.8973 (16) | C18—N8 | 1.425 (3) |
| N3—H3n | 0.8624 (18) | H18a—H18b | 1.5677 |
| N3—H4n | 0.8630 (15) | H18a—H18c | 1.5677 |
| N5—H5n | 0.8658 (14) | H18b—H18c | 1.5677 |
| N5—H6n | 0.8630 (16) | C19—H19a | 0.96 |
| C10—C11 | 1.503 (3) | C19—H19b | 0.96 |
| C10—O1 | 1.267 (3) | C19—H19c | 0.96 |
| C10—O2 | 1.231 (2) | C19—N8 | 1.470 (3) |
| C11—C12 | 1.405 (2) | H19a—H19b | 1.5677 |
| C11—C16 | 1.382 (3) | H19a—H19c | 1.5677 |
| C12—C13 | 1.402 (3) | H19b—H19c | 1.5677 |
| C12—O3 | 1.321 (3) | | |
| | | | |
| C2—C1—C6 | 120.20 (18) | C11—C12—O3 | 122.09 (17) |
| C2—C1—C7 | 122.86 (19) | C13—C12—O3 | 120.57 (17) |
| C6—C1—C7 | 116.9 (2) | C12—C13—C14 | 122.17 (18) |
| C1—C2—C3 | 120.2 (2) | C12—C13—N6 | 118.7 (2) |

| | | | |
|---|---|---|---|
| C2—C3—C4 | 119.6 (2) | C14—C13—N6 | 119.10 (19) |
| C3—C4—H4 | 120.02 | C13—C14—H14 | 120.79 |
| C3—C4—C5 | 120.0 (2) | C13—C14—C15 | 118.4 (2) |
| H4—C4—C5 | 120.02 | H14—C14—C15 | 120.79 |
| C4—C5—H5 | 118.63 | C14—C15—C16 | 122.1 (2) |
| C4—C5—C6 | 122.7 (2) | C14—C15—N7 | 119.4 (2) |
| H5—C5—C6 | 118.63 | C16—C15—N7 | 118.5 (2) |
| C1—C6—C5 | 117.3 (2) | C11—C16—C15 | 119.19 (18) |
| C1—C6—H6 | 121.37 | C11—C16—H16 | 120.41 |
| C5—C6—H6 | 121.36 | C15—C16—H16 | 120.41 |
| C1—C7—C9 | 124.50 (16) | C13—N6—O4 | 118.2 (2) |
| C1—C7—N1 | 115.60 (15) | C13—N6—O5 | 117.0 (3) |
| C9—C7—N1 | 119.58 (16) | O4—N6—O5 | 124.8 (2) |
| N2—C8—N3 | 118.61 (17) | C15—N7—O6 | 116.6 (2) |
| N2—C8—N4 | 120.57 (17) | C15—N7—O7 | 118.1 (3) |
| N3—C8—N4 | 120.81 (16) | O6—N7—O7 | 125.3 (3) |
| C7—C9—N4 | 120.53 (16) | C10—O1—H3o | 101.27 (12) |
| C7—C9—N5 | 120.93 (16) | C12—O3—H3o | 99.28 (14) |
| N4—C9—N5 | 118.52 (15) | H17—C17—N8 | 116.69 |
| C7—N1—N2 | 117.85 (14) | H17—C17—O8 | 116.69 |
| C8—N2—N1 | 123.77 (16) | N8—C17—O8 | 126.6 (2) |
| C8—N2—H2n | 122.42 (17) | H18a—C18—H18b | 109.47 |
| N1—N2—H2n | 113.82 (14) | H18a—C18—H18c | 109.47 |
| C8—N3—H3n | 122.28 (17) | H18a—C18—N8 | 109.47 |
| C8—N3—H4n | 121.07 (18) | H18b—C18—H18c | 109.47 |
| H3n—N3—H4n | 116.2 (2) | H18b—C18—N8 | 109.47 |
| C8—N4—C9 | 117.69 (14) | H18c—C18—N8 | 109.47 |
| C9—N5—H5n | 119.55 (17) | H19a—C19—H19b | 109.47 |
| C9—N5—H6n | 124.81 (16) | H19a—C19—H19c | 109.47 |
| H5n—N5—H6n | 115.64 (18) | H19a—C19—N8 | 109.47 |
| C11—C10—O1 | 115.76 (15) | H19b—C19—H19c | 109.47 |
| C11—C10—O2 | 119.27 (19) | H19b—C19—N8 | 109.47 |
| O1—C10—O2 | 124.96 (19) | H19c—C19—N8 | 109.47 |
| C10—C11—C12 | 119.51 (18) | C17—N8—C18 | 120.7 (2) |
| C10—C11—C16 | 119.71 (16) | C17—N8—C19 | 119.5 (2) |
| C12—C11—C16 | 120.74 (18) | C18—N8—C19 | 119.6 (2) |
| C11—C12—C13 | 117.3 (2) | O1—H3o—O3 | 161.33 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C19—H19*a*···O4$^{i}$ | 0.96 | 2.47 | 3.401 (4) | 163 |
| N3—H3*n*···O2 | 0.8624 (18) | 1.9939 (16) | 2.854 (2) | 174.78 (11) |
| N3—H4*n*···O8$^{ii}$ | 0.8630 (15) | 2.0586 (14) | 2.921 (2) | 176.91 (12) |
| N2—H2*n*···O1 | 0.8973 (16) | 1.8310 (15) | 2.728 (2) | 177.45 (12) |
| N5—H5*n*···N4$^{iii}$ | 0.8658 (14) | 2.1409 (13) | 2.9992 (19) | 171.06 (11) |

| | | | | |
|---|---|---|---|---|
| N5—H6*n*···O8$^{iv}$ | 0.8630 (16) | 2.0412 (16) | 2.760 (2) | 140.22 (10) |
| O3—H3*o*···O1 | 0.9258 (14) | 1.5621 (12) | 2.4572 (18) | 161.33 (12) |

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

**Bis(1,10-phenanthroline-5,6-dione-$\kappa^2N,N'$)silver(I) 3,5-dinitrosalicylate (SAFGUD)**

*Crystal data*

$[Ag(C_{12}H_6N_2O_2)](C_7H_3N_2O_7)$
$M_r$ = 755.36
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
*a* = 11.757 (2) Å
*b* = 18.297 (4) Å
*c* = 13.223 (3) Å
$\beta$ = 103.91 (3)°
*V* = 2761.1 (11) Å$^3$
*Z* = 4
*F*(000) = 1512
$D_x$ = 1.817 Mg m$^{-3}$
Mo *K*$\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 5197 reflections
$\theta$ = 3.2–25.4°
$\mu$ = 0.81 mm$^{-1}$
*T* = 174 K
Prism, yellow
0.3 × 0.24 × 0.2 mm

*Data collection*

Oxford Diffraction Gemini R Ultra diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min}$ = 0.780, $T_{max}$ = 0.910
12726 measured reflections
5013 independent reflections
3100 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.052
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 3.2°
*h* = −14→11
*k* = −17→22
*l* = −15→13

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)]$ = 0.062
$wR(F)$ = 0.118
*S* = 1.64
5013 reflections
444 parameters
0 restraints
56 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.016
$\Delta\rho_{max}$ = 0.76 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.63 e Å$^{-3}$
Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 2400 (800)

*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**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
Number of fixed parameters 3

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Ag1 | 0.21070 (4) | 0.44031 (3) | 0.52479 (4) | 0.0538 (2) |
| C1 | 0.4796 (5) | 0.5138 (3) | 0.6221 (4) | 0.043 (2) |
| H1 | 0.440696 | 0.558209 | 0.60689 | 0.0511* |
| C2 | 0.5975 (6) | 0.5153 (3) | 0.6692 (4) | 0.046 (2) |
| H2 | 0.637009 | 0.559483 | 0.684873 | 0.0553* |
| C3 | 0.6551 (5) | 0.4500 (3) | 0.6925 (4) | 0.046 (2) |
| H3 | 0.734391 | 0.449118 | 0.725729 | 0.0547* |
| C4 | 0.5946 (5) | 0.3859 (3) | 0.6661 (4) | 0.035 (2) |
| C5 | 0.6523 (8) | 0.3141 (4) | 0.6884 (5) | 0.068 (3) |
| C6 | 0.5912 (10) | 0.2510 (4) | 0.6718 (6) | 0.093 (5) |
| C7 | 0.4628 (6) | 0.2534 (3) | 0.6228 (4) | 0.042 (2) |
| C8 | 0.3994 (7) | 0.1881 (3) | 0.6033 (4) | 0.052 (3) |
| H8 | 0.435089 | 0.143184 | 0.6224 | 0.0623* |
| C9 | 0.2837 (7) | 0.1928 (4) | 0.5555 (5) | 0.063 (3) |
| H9 | 0.238665 | 0.150616 | 0.540161 | 0.0756* |
| C10 | 0.2345 (6) | 0.2592 (3) | 0.5304 (5) | 0.052 (3) |
| H10 | 0.155582 | 0.26107 | 0.496188 | 0.0629* |
| C11 | 0.4065 (5) | 0.3197 (3) | 0.5966 (4) | 0.032 (2) |
| C12 | 0.4742 (5) | 0.3885 (3) | 0.6197 (4) | 0.033 (2) |
| C13 | 0.0851 (5) | 0.4150 (3) | 0.2774 (5) | 0.054 (3) |
| H13 | 0.109147 | 0.367048 | 0.293174 | 0.0649* |
| C14 | 0.0376 (5) | 0.4324 (4) | 0.1743 (5) | 0.060 (3) |
| H14 | 0.029151 | 0.397085 | 0.122358 | 0.0717* |
| C15 | 0.0032 (5) | 0.5033 (3) | 0.1508 (5) | 0.051 (3) |
| H15 | −0.030371 | 0.516779 | 0.082274 | 0.0617* |
| C16 | 0.0190 (5) | 0.5550 (3) | 0.2306 (4) | 0.040 (2) |
| C17 | −0.0145 (5) | 0.6317 (3) | 0.2063 (5) | 0.051 (3) |
| C18 | −0.0094 (5) | 0.6826 (3) | 0.2940 (5) | 0.053 (3) |
| C19 | 0.0474 (5) | 0.6578 (3) | 0.4023 (5) | 0.042 (2) |
| C20 | 0.0689 (5) | 0.7058 (3) | 0.4858 (5) | 0.052 (3) |
| H20 | 0.048099 | 0.754726 | 0.475014 | 0.063* |
| C21 | 0.1204 (6) | 0.6817 (4) | 0.5833 (5) | 0.058 (3) |
| H21 | 0.134626 | 0.713123 | 0.64029 | 0.0692* |
| C22 | 0.1503 (5) | 0.6091 (4) | 0.5941 (5) | 0.054 (3) |
| H22 | 0.186352 | 0.59254 | 0.660553 | 0.0653* |
| C23 | 0.0818 (5) | 0.5847 (3) | 0.4195 (4) | 0.036 (2) |
| C24 | 0.0637 (4) | 0.5318 (3) | 0.3326 (4) | 0.032 (2) |
| C25 | 0.6150 (6) | 0.4142 (3) | 0.9276 (4) | 0.041 (2) |
| C26 | 0.5079 (6) | 0.4527 (3) | 0.8869 (4) | 0.040 (2) |
| C27 | 0.4074 (5) | 0.4101 (3) | 0.8426 (4) | 0.037 (2) |
| C28 | 0.4091 (5) | 0.3351 (3) | 0.8456 (4) | 0.041 (2) |
| H28 | 0.341998 | 0.308064 | 0.817616 | 0.0498* |
| C29 | 0.5156 (6) | 0.3004 (3) | 0.8924 (4) | 0.041 (2) |
| C30 | 0.6177 (5) | 0.3387 (3) | 0.9323 (4) | 0.043 (2) |
| H30 | 0.687051 | 0.314123 | 0.961804 | 0.0515* |

| | | | | |
|---|---|---|---|---|
| C31 | 0.2956 (6) | 0.4467 (4) | 0.7881 (5) | 0.047 (3) |
| N1 | 0.4179 (4) | 0.4525 (2) | 0.5968 (3) | 0.0338 (17) |
| N2 | 0.2929 (4) | 0.3229 (2) | 0.5519 (3) | 0.0387 (18) |
| N3 | 0.0989 (4) | 0.4627 (2) | 0.3563 (3) | 0.0413 (18) |
| N4 | 0.1317 (4) | 0.5603 (2) | 0.5163 (3) | 0.0405 (18) |
| N5 | 0.7267 (6) | 0.4510 (4) | 0.9628 (4) | 0.059 (3) |
| N6 | 0.5168 (6) | 0.2209 (3) | 0.9002 (4) | 0.055 (3) |
| O1 | 0.7646 (5) | 0.3117 (3) | 0.7273 (4) | 0.092 (3) |
| O2 | 0.6406 (5) | 0.1892 (3) | 0.6987 (4) | 0.102 (3) |
| O3 | −0.0421 (4) | 0.6544 (2) | 0.1169 (3) | 0.069 (2) |
| O4 | −0.0501 (5) | 0.7444 (2) | 0.2776 (4) | 0.081 (2) |
| O5 | 0.8168 (5) | 0.4145 (3) | 0.9750 (4) | 0.090 (3) |
| O6 | 0.7298 (5) | 0.5169 (3) | 0.9762 (4) | 0.084 (2) |
| O7 | 0.5025 (4) | 0.5233 (2) | 0.8850 (3) | 0.0617 (19) |
| O8 | 0.2936 (4) | 0.5168 (3) | 0.7945 (3) | 0.066 (2) |
| O9 | 0.2137 (4) | 0.4111 (3) | 0.7370 (3) | 0.064 (2) |
| O10 | 0.4286 (5) | 0.1877 (2) | 0.8580 (4) | 0.070 (2) |
| O11 | 0.6096 (5) | 0.1916 (2) | 0.9512 (4) | 0.068 (2) |
| H7 | 0.388884 | 0.531747 | 0.83591 | 0.019 (12)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ag1 | 0.0474 (4) | 0.0492 (3) | 0.0603 (3) | 0.0150 (3) | 0.0038 (2) | 0.0149 (3) |
| C1 | 0.054 (5) | 0.035 (4) | 0.041 (3) | −0.006 (3) | 0.014 (3) | 0.001 (3) |
| C2 | 0.052 (5) | 0.041 (4) | 0.048 (4) | −0.010 (3) | 0.018 (3) | −0.001 (3) |
| C3 | 0.031 (4) | 0.063 (4) | 0.043 (3) | −0.004 (3) | 0.008 (3) | −0.008 (3) |
| C4 | 0.036 (4) | 0.034 (3) | 0.035 (3) | 0.015 (3) | 0.013 (3) | 0.004 (3) |
| C5 | 0.085 (7) | 0.078 (6) | 0.048 (4) | 0.014 (5) | 0.031 (4) | 0.006 (4) |
| C6 | 0.201 (12) | 0.037 (5) | 0.065 (5) | 0.012 (6) | 0.081 (7) | 0.001 (4) |
| C7 | 0.033 (4) | 0.059 (5) | 0.035 (3) | 0.007 (3) | 0.008 (3) | 0.001 (3) |
| C8 | 0.088 (6) | 0.023 (3) | 0.051 (4) | 0.010 (4) | 0.029 (4) | 0.005 (3) |
| C9 | 0.074 (6) | 0.053 (5) | 0.067 (5) | −0.009 (4) | 0.025 (4) | −0.008 (4) |
| C10 | 0.039 (4) | 0.054 (4) | 0.064 (4) | −0.003 (4) | 0.012 (3) | −0.014 (4) |
| C11 | 0.043 (4) | 0.026 (3) | 0.029 (3) | 0.006 (3) | 0.010 (3) | 0.002 (2) |
| C12 | 0.043 (4) | 0.031 (3) | 0.028 (3) | −0.004 (3) | 0.014 (3) | 0.002 (2) |
| C13 | 0.052 (5) | 0.034 (4) | 0.071 (5) | −0.002 (3) | 0.005 (4) | −0.001 (3) |
| C14 | 0.055 (5) | 0.055 (5) | 0.064 (4) | 0.000 (4) | 0.003 (4) | −0.024 (4) |
| C15 | 0.046 (4) | 0.057 (4) | 0.045 (4) | −0.004 (3) | 0.000 (3) | −0.002 (3) |
| C16 | 0.029 (3) | 0.038 (3) | 0.051 (4) | 0.000 (3) | 0.004 (3) | 0.005 (3) |
| C17 | 0.039 (4) | 0.054 (4) | 0.055 (4) | 0.002 (3) | 0.001 (3) | 0.021 (4) |
| C18 | 0.036 (4) | 0.042 (4) | 0.077 (5) | −0.003 (3) | 0.004 (3) | 0.005 (4) |
| C19 | 0.035 (4) | 0.032 (3) | 0.057 (4) | 0.003 (3) | 0.007 (3) | 0.002 (3) |
| C20 | 0.046 (4) | 0.031 (3) | 0.079 (5) | 0.000 (3) | 0.012 (4) | −0.010 (4) |
| C21 | 0.049 (5) | 0.055 (5) | 0.067 (5) | 0.002 (4) | 0.010 (4) | −0.013 (4) |
| C22 | 0.046 (4) | 0.070 (5) | 0.042 (4) | −0.003 (4) | 0.001 (3) | −0.006 (4) |
| C23 | 0.027 (3) | 0.034 (3) | 0.046 (3) | 0.002 (3) | 0.006 (3) | 0.000 (3) |
| C24 | 0.019 (3) | 0.031 (3) | 0.046 (3) | −0.001 (2) | 0.007 (2) | 0.004 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C25 | 0.058 (5) | 0.037 (4) | 0.028 (3) | −0.010 (3) | 0.010 (3) | −0.004 (3) |
| C26 | 0.062 (5) | 0.032 (3) | 0.030 (3) | 0.001 (3) | 0.018 (3) | −0.002 (3) |
| C27 | 0.039 (4) | 0.038 (4) | 0.035 (3) | 0.002 (3) | 0.012 (3) | 0.006 (3) |
| C28 | 0.057 (5) | 0.041 (4) | 0.030 (3) | −0.007 (3) | 0.018 (3) | −0.002 (3) |
| C29 | 0.068 (5) | 0.029 (3) | 0.032 (3) | 0.004 (3) | 0.024 (3) | 0.005 (3) |
| C30 | 0.047 (4) | 0.047 (4) | 0.036 (3) | 0.005 (3) | 0.014 (3) | 0.002 (3) |
| C31 | 0.057 (5) | 0.041 (4) | 0.048 (4) | 0.011 (4) | 0.023 (3) | 0.008 (3) |
| N1 | 0.036 (3) | 0.031 (3) | 0.033 (2) | 0.005 (2) | 0.008 (2) | 0.001 (2) |
| N2 | 0.033 (3) | 0.037 (3) | 0.044 (3) | −0.001 (2) | 0.006 (2) | −0.002 (2) |
| N3 | 0.040 (3) | 0.029 (3) | 0.051 (3) | 0.001 (2) | 0.004 (2) | −0.003 (2) |
| N4 | 0.035 (3) | 0.043 (3) | 0.041 (3) | 0.003 (2) | 0.004 (2) | 0.006 (2) |
| N5 | 0.057 (4) | 0.072 (5) | 0.044 (3) | −0.015 (4) | 0.003 (3) | −0.009 (3) |
| N6 | 0.090 (5) | 0.039 (4) | 0.044 (3) | 0.004 (3) | 0.033 (3) | 0.004 (3) |
| O1 | 0.083 (4) | 0.104 (4) | 0.088 (4) | 0.034 (4) | 0.022 (3) | 0.021 (3) |
| O2 | 0.095 (5) | 0.085 (4) | 0.115 (5) | 0.014 (4) | 0.002 (4) | −0.007 (4) |
| O3 | 0.066 (3) | 0.071 (3) | 0.065 (3) | 0.002 (3) | 0.004 (3) | 0.026 (3) |
| O4 | 0.101 (4) | 0.040 (3) | 0.095 (4) | 0.019 (3) | 0.007 (3) | 0.017 (3) |
| O5 | 0.052 (4) | 0.100 (4) | 0.111 (4) | −0.002 (3) | 0.006 (3) | −0.031 (3) |
| O6 | 0.091 (4) | 0.051 (3) | 0.090 (4) | −0.024 (3) | −0.015 (3) | 0.003 (3) |
| O7 | 0.088 (4) | 0.042 (3) | 0.057 (3) | −0.005 (2) | 0.021 (3) | −0.002 (2) |
| O8 | 0.069 (4) | 0.060 (3) | 0.072 (3) | 0.017 (3) | 0.022 (3) | 0.009 (2) |
| O9 | 0.042 (3) | 0.084 (4) | 0.066 (3) | 0.006 (3) | 0.012 (2) | −0.002 (3) |
| O10 | 0.107 (5) | 0.039 (3) | 0.067 (3) | −0.016 (3) | 0.025 (3) | −0.001 (2) |
| O11 | 0.089 (4) | 0.042 (3) | 0.081 (3) | 0.016 (3) | 0.036 (3) | 0.022 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ag1—N1 | 2.404 (4) | C16—C24 | 1.392 (7) |
| Ag1—N2 | 2.348 (5) | C17—C18 | 1.476 (9) |
| Ag1—N3 | 2.335 (4) | C17—O3 | 1.220 (8) |
| Ag1—N4 | 2.376 (5) | C18—C19 | 1.498 (8) |
| C1—H1 | 0.93 | C18—O4 | 1.227 (8) |
| C1—C2 | 1.376 (8) | C19—C20 | 1.386 (8) |
| C1—N1 | 1.334 (7) | C19—C23 | 1.400 (8) |
| C2—H2 | 0.93 | C20—H20 | 0.93 |
| C2—C3 | 1.373 (8) | C20—C21 | 1.359 (9) |
| C3—H3 | 0.93 | C21—H21 | 0.93 |
| C3—C4 | 1.372 (8) | C21—C22 | 1.373 (9) |
| C4—C5 | 1.475 (10) | C22—H22 | 0.93 |
| C4—C12 | 1.402 (8) | C22—N4 | 1.341 (8) |
| C5—C6 | 1.349 (12) | C23—C24 | 1.478 (7) |
| C5—O1 | 1.298 (10) | C23—N4 | 1.349 (7) |
| C6—C7 | 1.493 (13) | C24—N3 | 1.344 (7) |
| C6—O2 | 1.283 (10) | C25—C26 | 1.430 (9) |
| C7—C8 | 1.399 (9) | C25—C30 | 1.384 (8) |
| C7—C11 | 1.385 (8) | C25—N5 | 1.450 (9) |
| C8—H8 | 0.93 | C26—C27 | 1.418 (8) |
| C8—C9 | 1.357 (10) | C26—O7 | 1.293 (7) |

| | | | |
|---|---|---|---|
| C9—H9 | 0.93 | C27—C28 | 1.373 (8) |
| C9—C10 | 1.353 (9) | C27—C31 | 1.496 (8) |
| C10—H10 | 0.93 | C28—H28 | 0.93 |
| C10—N2 | 1.347 (8) | C28—C29 | 1.407 (8) |
| C11—C12 | 1.481 (7) | C29—C30 | 1.380 (8) |
| C11—N2 | 1.326 (7) | C29—N6 | 1.458 (8) |
| C12—N1 | 1.344 (7) | C30—H30 | 0.93 |
| C13—H13 | 0.93 | C31—O8 | 1.286 (8) |
| C13—C14 | 1.381 (9) | C31—O9 | 1.223 (8) |
| C13—N3 | 1.339 (8) | N5—O5 | 1.230 (9) |
| C14—H14 | 0.93 | N5—O6 | 1.218 (8) |
| C14—C15 | 1.373 (9) | N6—O10 | 1.216 (8) |
| C15—H15 | 0.93 | N6—O11 | 1.257 (8) |
| C15—C16 | 1.396 (8) | O7—H7 | 1.346 (4) |
| C16—C17 | 1.473 (8) | O8—H7 | 1.155 (4) |

| | | | |
|---|---|---|---|
| H1—C1—C2 | 118.04 | C19—C18—O4 | 120.9 (6) |
| H1—C1—N1 | 118.04 | C18—C19—C20 | 121.6 (5) |
| C2—C1—N1 | 123.9 (5) | C18—C19—C23 | 119.3 (5) |
| C1—C2—H2 | 120.88 | C20—C19—C23 | 119.1 (5) |
| C1—C2—C3 | 118.2 (5) | C19—C20—H20 | 119.84 |
| H2—C2—C3 | 120.88 | C19—C20—C21 | 120.3 (6) |
| C2—C3—H3 | 120.34 | H20—C20—C21 | 119.84 |
| C2—C3—C4 | 119.3 (5) | C20—C21—H21 | 121.42 |
| H3—C3—C4 | 120.34 | C20—C21—C22 | 117.2 (6) |
| C3—C4—C5 | 121.7 (6) | H21—C21—C22 | 121.42 |
| C3—C4—C12 | 119.3 (5) | C21—C22—H22 | 117.46 |
| C5—C4—C12 | 119.0 (5) | C21—C22—N4 | 125.1 (5) |
| C4—C5—C6 | 121.9 (8) | H22—C22—N4 | 117.46 |
| C4—C5—O1 | 118.9 (7) | C19—C23—C24 | 121.2 (5) |
| C6—C5—O1 | 119.2 (8) | C19—C23—N4 | 120.8 (5) |
| C5—C6—C7 | 119.3 (7) | C24—C23—N4 | 118.0 (5) |
| C5—C6—O2 | 121.4 (9) | C16—C24—C23 | 120.2 (5) |
| C7—C6—O2 | 119.3 (7) | C16—C24—N3 | 122.5 (5) |
| C6—C7—C8 | 119.6 (6) | C23—C24—N3 | 117.2 (4) |
| C6—C7—C11 | 120.4 (6) | C26—C25—C30 | 121.2 (5) |
| C8—C7—C11 | 120.0 (6) | C26—C25—N5 | 122.7 (5) |
| C7—C8—H8 | 121.23 | C30—C25—N5 | 116.1 (6) |
| C7—C8—C9 | 117.5 (6) | C25—C26—C27 | 117.1 (5) |
| H8—C8—C9 | 121.23 | C25—C26—O7 | 122.2 (5) |
| C8—C9—H9 | 120.26 | C27—C26—O7 | 120.5 (5) |
| C8—C9—C10 | 119.5 (6) | C26—C27—C28 | 122.1 (5) |
| H9—C9—C10 | 120.26 | C26—C27—C31 | 120.1 (5) |
| C9—C10—H10 | 118.01 | C28—C27—C31 | 117.8 (5) |
| C9—C10—N2 | 124.0 (6) | C27—C28—H28 | 121 |
| H10—C10—N2 | 118.01 | C27—C28—C29 | 118.0 (5) |
| C7—C11—C12 | 119.5 (5) | H28—C28—C29 | 121 |
| C7—C11—N2 | 121.3 (5) | C28—C29—C30 | 122.6 (5) |

| | | | |
|---|---|---|---|
| C12—C11—N2 | 119.2 (5) | C28—C29—N6 | 118.3 (5) |
| C4—C12—C11 | 119.8 (5) | C30—C29—N6 | 119.1 (5) |
| C4—C12—N1 | 121.2 (5) | C25—C30—C29 | 118.7 (5) |
| C11—C12—N1 | 118.9 (5) | C25—C30—H30 | 120.65 |
| H13—C13—C14 | 117.83 | C29—C30—H30 | 120.65 |
| H13—C13—N3 | 117.83 | C27—C31—O8 | 116.2 (5) |
| C14—C13—N3 | 124.3 (5) | C27—C31—O9 | 120.8 (6) |
| C13—C14—H14 | 121.04 | O8—C31—O9 | 122.9 (6) |
| C13—C14—C15 | 117.9 (6) | C1—N1—C12 | 117.9 (4) |
| H14—C14—C15 | 121.04 | C10—N2—C11 | 117.6 (5) |
| C14—C15—H15 | 120.29 | C13—N3—C24 | 117.3 (5) |
| C14—C15—C16 | 119.4 (5) | C22—N4—C23 | 117.5 (5) |
| H15—C15—C16 | 120.29 | C25—N5—O5 | 118.4 (6) |
| C15—C16—C17 | 120.1 (5) | C25—N5—O6 | 120.0 (6) |
| C15—C16—C24 | 118.5 (5) | O5—N5—O6 | 121.6 (6) |
| C17—C16—C24 | 121.4 (5) | C29—N6—O10 | 118.3 (5) |
| C16—C17—C18 | 118.1 (5) | C29—N6—O11 | 117.2 (5) |
| C16—C17—O3 | 122.0 (6) | O10—N6—O11 | 124.5 (5) |
| C18—C17—O3 | 119.9 (6) | C26—O7—H7 | 99.4 (4) |
| C17—C18—C19 | 119.0 (5) | C31—O8—H7 | 103.7 (4) |
| C17—C18—O4 | 120.1 (6) | O7—H7—O8 | 159.6 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2···O10$^{i}$ | 0.93 | 2.49 | 3.180 (7) | 131 |
| C8—H8···O7$^{ii}$ | 0.93 | 2.32 | 3.220 (7) | 162 |
| C9—H9···O6$^{ii}$ | 0.93 | 2.49 | 3.243 (9) | 138 |
| C13—H13···O4$^{iii}$ | 0.93 | 2.47 | 3.209 (7) | 137 |
| C22—H22···O8 | 0.93 | 2.36 | 3.251 (7) | 160 |
| O7—H7···C31 | 1.346 (4) | 1.922 (7) | 2.833 (8) | 119.1 (3) |
| O7—H7···O8 | 1.346 (4) | 1.155 (4) | 2.462 (6) | 159.6 (3) |
| O8—H7···C26 | 1.155 (4) | 2.013 (6) | 2.781 (7) | 120.3 (3) |
| O8—H7···O7 | 1.155 (4) | 1.346 (4) | 2.462 (6) | 159.6 (3) |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) −*x*, *y*−1/2, −*z*+1/2.

### 3,5-Dimethylpyrazolium 3,5-dinitrosalicylate (SEDKET)

*Crystal data*

$C_5H_9N_2^+·C_7H_3N_2O_7^-$
$M_r$ = 324.26
Monoclinic, $P2_1$
*a* = 8.1183 (7) Å
*b* = 6.0636 (5) Å
*c* = 14.1453 (11) Å
*β* = 91.904 (1)°
*V* = 695.93 (10) Å$^3$
*Z* = 2
*F*(000) = 336
$D_x$ = 1.547 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1025 reflections
*θ* = 2.5–22.6°
*μ* = 0.13 mm$^{-1}$
*T* = 293 K
Block, colorless
0.40 × 0.27 × 0.11 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)
$T_{min}$ = 0.959, $T_{max}$ = 0.986
3523 measured reflections
2301 independent reflections
1444 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.040
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.5°
$h$ = −9→9
$k$ = −7→7
$l$ = −16→12

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)]$ = 0.041
$wR(F)$ = 0.088
$S$ = 1.16
2301 reflections
212 parameters
0 restraints
37 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.033
$\Delta\rho_{max}$ = 0.11 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.10 e Å$^{-3}$
Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 3100 (400)
Absolute structure: 955 of Friedel pairs used in the refinement
Absolute structure parameter: 0.5

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger. Number of fixed parameters 10.

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.2262 (4) | 0.7164 (5) | 0.2338 (2) | 0.0464 (11) |
| N2 | 0.2203 (3) | 0.6196 (5) | 0.3198 (2) | 0.0478 (11) |
| C6 | 0.3519 (4) | 0.7226 (6) | 0.5525 (2) | 0.0412 (12) |
| N3 | 0.2179 (3) | 1.3158 (5) | 0.7705 (2) | 0.0509 (12) |
| N4 | 0.5552 (4) | 0.7120 (5) | 0.8913 (2) | 0.0501 (12) |
| O1 | 0.5417 (3) | 0.5285 (4) | 0.70239 (16) | 0.0462 (9) |
| O2 | 0.4266 (3) | 0.5357 (4) | 0.53811 (15) | 0.0582 (10) |
| O3 | 0.2657 (3) | 0.8100 (4) | 0.49006 (14) | 0.0507 (8) |
| O4 | 0.1415 (3) | 1.4013 (4) | 0.70419 (18) | 0.0652 (10) |
| O5 | 0.2270 (3) | 1.3968 (4) | 0.85025 (18) | 0.0709 (11) |
| O6 | 0.5263 (4) | 0.7839 (5) | 0.96960 (17) | 0.0853 (12) |
| O7 | 0.6549 (4) | 0.5664 (5) | 0.87980 (17) | 0.0719 (12) |

| | | | | |
|---|---|---|---|---|
| C1 | 0.1300 (5) | 0.6431 (8) | 0.0685 (2) | 0.0762 (19) |
| H1a | 0.132119 | 0.800298 | 0.060733 | 0.1144* |
| H1b | 0.027525 | 0.586015 | 0.043068 | 0.1144* |
| H1c | 0.219699 | 0.578396 | 0.035744 | 0.1144* |
| C2 | 0.1466 (4) | 0.5878 (6) | 0.1713 (2) | 0.0469 (13) |
| C3 | 0.0892 (4) | 0.4058 (7) | 0.2187 (3) | 0.0553 (14) |
| H3 | 0.029651 | 0.288588 | 0.192446 | 0.0663* |
| C4 | 0.1373 (4) | 0.4313 (6) | 0.3133 (2) | 0.0481 (13) |
| C5 | 0.1093 (5) | 0.2896 (7) | 0.3966 (3) | 0.0669 (16) |
| H5a | 0.025142 | 0.183251 | 0.380914 | 0.1003* |
| H5b | 0.20964 | 0.214551 | 0.414477 | 0.1003* |
| H5c | 0.074882 | 0.379302 | 0.448238 | 0.1003* |
| C12 | 0.4691 (4) | 0.7102 (6) | 0.7196 (2) | 0.0372 (12) |
| C7 | 0.3736 (3) | 0.8233 (6) | 0.6477 (2) | 0.0348 (11) |
| C8 | 0.2972 (4) | 1.0181 (6) | 0.6641 (2) | 0.0380 (12) |
| H8 | 0.239805 | 1.089516 | 0.615013 | 0.0456* |
| C9 | 0.3044 (4) | 1.1108 (6) | 0.7534 (2) | 0.0381 (11) |
| C10 | 0.3902 (4) | 1.0091 (6) | 0.8274 (2) | 0.0403 (12) |
| H10 | 0.393751 | 1.071121 | 0.887603 | 0.0483* |
| C11 | 0.4704 (4) | 0.8133 (6) | 0.8100 (2) | 0.0380 (11) |
| H2a | 0.507 (5) | 0.485 (9) | 0.620 (3) | 0.145 (19)* |
| H1 | 0.289 (6) | 0.878 (9) | 0.225 (3) | 0.130 (19)* |
| H2 | 0.258 (4) | 0.707 (6) | 0.373 (2) | 0.063 (12)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0502 (18) | 0.049 (2) | 0.0399 (18) | 0.0006 (15) | −0.0027 (14) | −0.0034 (15) |
| N2 | 0.0497 (19) | 0.051 (2) | 0.0422 (18) | −0.0046 (17) | −0.0047 (14) | −0.0066 (17) |
| C6 | 0.0393 (19) | 0.047 (2) | 0.037 (2) | −0.0016 (18) | 0.0053 (16) | −0.0027 (18) |
| N3 | 0.0500 (19) | 0.043 (2) | 0.060 (2) | −0.0003 (17) | 0.0048 (16) | −0.0031 (18) |
| N4 | 0.065 (2) | 0.046 (2) | 0.0387 (19) | 0.0015 (17) | −0.0037 (16) | 0.0042 (16) |
| O1 | 0.0564 (15) | 0.0414 (15) | 0.0407 (14) | 0.0091 (13) | −0.0027 (11) | −0.0072 (12) |
| O2 | 0.0704 (17) | 0.0626 (18) | 0.0414 (14) | 0.0205 (15) | −0.0036 (12) | −0.0156 (14) |
| O3 | 0.0581 (14) | 0.0600 (17) | 0.0333 (13) | 0.0050 (14) | −0.0083 (11) | 0.0013 (12) |
| O4 | 0.0703 (17) | 0.0521 (17) | 0.0721 (18) | 0.0185 (15) | −0.0141 (14) | 0.0031 (15) |
| O5 | 0.092 (2) | 0.0593 (19) | 0.0617 (17) | 0.0072 (16) | 0.0048 (14) | −0.0187 (15) |
| O6 | 0.148 (3) | 0.075 (2) | 0.0326 (16) | 0.035 (2) | −0.0069 (15) | −0.0032 (15) |
| O7 | 0.092 (2) | 0.072 (2) | 0.0510 (16) | 0.0308 (19) | −0.0083 (14) | 0.0090 (16) |
| C1 | 0.086 (3) | 0.096 (4) | 0.047 (2) | −0.014 (3) | −0.009 (2) | −0.007 (2) |
| C2 | 0.043 (2) | 0.054 (3) | 0.0434 (19) | 0.005 (2) | −0.0046 (17) | −0.009 (2) |
| C3 | 0.049 (2) | 0.057 (3) | 0.060 (2) | −0.004 (2) | −0.0075 (18) | −0.022 (2) |
| C4 | 0.039 (2) | 0.044 (2) | 0.062 (2) | 0.0027 (19) | 0.0037 (17) | −0.003 (2) |
| C5 | 0.066 (3) | 0.060 (3) | 0.074 (3) | 0.000 (2) | −0.002 (2) | 0.012 (2) |
| C12 | 0.0351 (19) | 0.041 (2) | 0.036 (2) | −0.0079 (18) | 0.0025 (15) | 0.0010 (17) |
| C7 | 0.0342 (17) | 0.039 (2) | 0.0316 (17) | −0.0052 (17) | 0.0004 (13) | 0.0031 (16) |
| C8 | 0.041 (2) | 0.038 (2) | 0.0349 (19) | −0.0032 (18) | −0.0036 (15) | 0.0039 (17) |
| C9 | 0.041 (2) | 0.032 (2) | 0.0416 (19) | −0.0045 (17) | 0.0020 (15) | −0.0015 (17) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.049 (2) | 0.039 (2) | 0.0325 (19) | −0.0038 (18) | 0.0020 (16) | −0.0017 (17) |
| C11 | 0.0419 (19) | 0.041 (2) | 0.0312 (17) | −0.0038 (19) | −0.0025 (14) | 0.0049 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—N2 | 1.353 (4) | H1a—H1b | 1.5677 |
| N1—C2 | 1.330 (5) | H1a—H1c | 1.5677 |
| N1—H1 | 1.11 (5) | H1b—H1c | 1.5677 |
| N2—C4 | 1.327 (5) | C2—C3 | 1.380 (5) |
| N2—H2 | 0.96 (3) | C3—H3 | 0.93 |
| C6—O2 | 1.304 (4) | C3—C4 | 1.389 (5) |
| C6—O3 | 1.229 (4) | C4—C5 | 1.482 (5) |
| C6—C7 | 1.484 (4) | C5—H5a | 0.96 |
| N3—O4 | 1.223 (4) | C5—H5b | 0.96 |
| N3—O5 | 1.231 (4) | C5—H5c | 0.96 |
| N3—C9 | 1.452 (5) | H5a—H5b | 1.5677 |
| N4—O6 | 1.220 (4) | H5a—H5c | 1.5677 |
| N4—O7 | 1.213 (4) | H5b—H5c | 1.5677 |
| N4—C11 | 1.457 (4) | C12—C7 | 1.433 (4) |
| O1—C12 | 1.277 (4) | C12—C11 | 1.422 (4) |
| O1—H2a | 1.22 (5) | C7—C8 | 1.358 (5) |
| O2—H2a | 1.34 (5) | C8—H8 | 0.93 |
| C1—H1a | 0.96 | C8—C9 | 1.382 (4) |
| C1—H1b | 0.96 | C9—C10 | 1.384 (4) |
| C1—H1c | 0.96 | C10—H10 | 0.93 |
| C1—C2 | 1.494 (5) | C10—C11 | 1.380 (5) |
| | | | |
| N2—N1—C2 | 108.2 (3) | N2—C4—C3 | 106.8 (3) |
| N2—N1—H1 | 121 (2) | N2—C4—C5 | 122.3 (3) |
| C2—N1—H1 | 131 (2) | C3—C4—C5 | 130.9 (3) |
| N1—N2—C4 | 110.1 (3) | C4—C5—H5a | 109.47 |
| N1—N2—H2 | 117 (2) | C4—C5—H5b | 109.47 |
| C4—N2—H2 | 132 (2) | C4—C5—H5c | 109.47 |
| O2—C6—O3 | 121.3 (3) | H5a—C5—H5b | 109.47 |
| O2—C6—C7 | 117.2 (3) | H5a—C5—H5c | 109.47 |
| O3—C6—C7 | 121.5 (3) | H5b—C5—H5c | 109.47 |
| O4—N3—O5 | 123.2 (3) | O1—C12—C7 | 121.3 (3) |
| O4—N3—C9 | 118.1 (3) | O1—C12—C11 | 124.1 (3) |
| O5—N3—C9 | 118.7 (3) | C7—C12—C11 | 114.5 (3) |
| O6—N4—O7 | 122.1 (3) | C6—C7—C12 | 119.5 (3) |
| O6—N4—C11 | 117.8 (3) | C6—C7—C8 | 118.1 (3) |
| O7—N4—C11 | 120.1 (3) | C12—C7—C8 | 122.3 (3) |
| C12—O1—H2a | 106 (2) | C7—C8—H8 | 119.87 |
| C6—O2—H2a | 106 (2) | C7—C8—C9 | 120.3 (3) |
| H1a—C1—H1b | 109.47 | H8—C8—C9 | 119.87 |
| H1a—C1—H1c | 109.47 | N3—C9—C8 | 119.6 (3) |
| H1a—C1—C2 | 109.47 | N3—C9—C10 | 119.3 (3) |
| H1b—C1—H1c | 109.47 | C8—C9—C10 | 121.0 (3) |

| | | | |
|---|---|---|---|
| H1b—C1—C2 | 109.47 | C9—C10—H10 | 120.77 |
| H1c—C1—C2 | 109.47 | C9—C10—C11 | 118.5 (3) |
| N1—C2—C1 | 122.8 (3) | H10—C10—C11 | 120.77 |
| N1—C2—C3 | 108.1 (3) | N4—C11—C12 | 120.9 (3) |
| C1—C2—C3 | 129.1 (3) | N4—C11—C10 | 115.7 (3) |
| C2—C3—H3 | 126.58 | C12—C11—C10 | 123.3 (3) |
| C2—C3—C4 | 106.8 (3) | O1—H2a—O2 | 149 (5) |
| H3—C3—C4 | 126.58 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C1—H1*a*···O7$^{i}$ | 0.96 | 2.49 | 3.176 (5) | 128 |
| C5—H5*a*···O4$^{ii}$ | 0.96 | 2.47 | 3.395 (5) | 162 |
| C10—H10···O6$^{iii}$ | 0.93 | 2.47 | 3.369 (4) | 164 |
| O1—H2*a*···O2 | 1.22 (5) | 1.34 (5) | 2.476 (3) | 149 (5) |
| O2—H2*a*···O1 | 1.34 (5) | 1.22 (5) | 2.476 (3) | 149 (5) |
| N1—H1···O1$^{i}$ | 1.11 (5) | 1.92 (5) | 2.799 (4) | 133 (3) |
| N1—H1···O7$^{i}$ | 1.11 (5) | 1.94 (5) | 2.850 (4) | 137 (3) |
| N2—H2···O3 | 0.96 (3) | 1.77 (3) | 2.685 (4) | 158 (3) |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1; (ii) −*x*, *y*−3/2, −*z*+1; (iii) −*x*+1, *y*+1/2, −*z*+2.

### 3-(1*H*-Imidazol-1-yl)propanaminium 2-carboxy-4,6-dinitrophenolate (TIYZIM)

*Crystal data*

$C_6H_{12}N_3^+·C_7H_3N_2O_7^-$
$M_r$ = 353.30
Triclinic, $P\overline{1}$
*a* = 7.0109 (4) Å
*b* = 10.6617 (8) Å
*c* = 10.7454 (7) Å
*α* = 93.075 (6)°
*β* = 95.863 (5)°
*γ* = 104.944 (6)°
*V* = 769.30 (9) Å$^3$
*Z* = 2
*F*(000) = 368
$D_x$ = 1.525 Mg m$^{-3}$
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 2218 reflections
*θ* = 4.2–72.3°
*μ* = 1.09 mm$^{-1}$
*T* = 173 K
Irregular, yellow
0.22 × 0.14 × 0.12 mm

*Data collection*

Agilent Xcalibur (Eos, Gemini) diffractometer
Graphite monochromator
Detector resolution: 16.0416 pixels mm$^{-1}$
*ω* scans
Absorption correction: multi-scan (CrysAlis PRO and CrysAlis RED; Agilent, 2012)
$T_{min}$ = 0.925, $T_{max}$ = 1.000
4664 measured reflections
2953 independent reflections
2426 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 72.5°, $\theta_{min}$ = 4.2°
*h* = −8→5
*k* = −12→13
*l* = −13→13

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)]$ = 0.041
*wR*(*F*) = 0.100
*S* = 1.64
2953 reflections

229 parameters
0 restraints
46 constraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement

Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max} = 0.013$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$
Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 740 (130)

*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**. Number of fixed parameters: 12

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1b | −0.19161 (17) | 0.67539 (12) | 0.52684 (11) | 0.0294 (4) |
| O2b | −0.38292 (17) | 0.47166 (12) | 0.40838 (12) | 0.0313 (4) |
| O3b | −0.25511 (17) | 0.37712 (12) | 0.26158 (12) | 0.0312 (4) |
| O4b | 0.41258 (19) | 0.58639 (13) | 0.16853 (13) | 0.0365 (5) |
| O5b | 0.59776 (18) | 0.75622 (13) | 0.28239 (14) | 0.0382 (5) |
| O6b | 0.3446 (2) | 0.93719 (14) | 0.62658 (15) | 0.0474 (5) |
| O7b | 0.02822 (19) | 0.91655 (13) | 0.61477 (13) | 0.0410 (5) |
| N1b | 0.1720 (2) | 0.88469 (14) | 0.58118 (14) | 0.0301 (5) |
| N2b | 0.4396 (2) | 0.67347 (14) | 0.25363 (14) | 0.0281 (5) |
| C1b | −0.0455 (2) | 0.68023 (16) | 0.46268 (15) | 0.0225 (5) |
| C2b | −0.0572 (2) | 0.57873 (15) | 0.36597 (15) | 0.0217 (5) |
| C3b | 0.0987 (2) | 0.57933 (16) | 0.29810 (15) | 0.0228 (5) |
| H3b | 0.086172 | 0.512562 | 0.233219 | 0.0273* |
| C4b | 0.2738 (2) | 0.67709 (16) | 0.32422 (15) | 0.0238 (5) |
| C5b | 0.2967 (2) | 0.77664 (16) | 0.41630 (15) | 0.0244 (5) |
| H5b | 0.418494 | 0.842618 | 0.433677 | 0.0292* |
| C6b | 0.1392 (2) | 0.77850 (16) | 0.48267 (15) | 0.0244 (5) |
| C7b | −0.2405 (2) | 0.46767 (16) | 0.33986 (15) | 0.0245 (5) |
| N1a | −0.2235 (2) | 0.05127 (14) | −0.17301 (14) | 0.0305 (5) |
| N2a | −0.0146 (2) | 0.22579 (13) | −0.06967 (13) | 0.0239 (4) |
| N3a | 0.3467 (2) | 0.20531 (13) | 0.28160 (13) | 0.0254 (4) |
| C1a | −0.0395 (2) | 0.12591 (16) | −0.15749 (16) | 0.0272 (6) |
| H1a | 0.06324 | 0.111094 | −0.202772 | 0.0327* |
| C2a | −0.3211 (3) | 0.10665 (17) | −0.08987 (17) | 0.0314 (6) |
| H2a | −0.457443 | 0.07432 | −0.079116 | 0.0377* |
| C3a | −0.1954 (3) | 0.21356 (17) | −0.02572 (17) | 0.0294 (6) |
| H3a | −0.225845 | 0.269084 | 0.03706 | 0.0353* |
| C4a | 0.1719 (2) | 0.32466 (17) | −0.02832 (15) | 0.0268 (5) |
| H4aa | 0.242014 | 0.351332 | −0.101965 | 0.0322* |
| H4ab | 0.142708 | 0.403798 | 0.008481 | 0.0322* |

| | | | | |
|---|---|---|---|---|
| C5a | 0.3076 (2) | 0.27759 (16) | 0.06668 (16) | 0.0271 (5) |
| H5aa | 0.324487 | 0.193134 | 0.033664 | 0.0326* |
| H5ab | 0.440667 | 0.340434 | 0.079114 | 0.0326* |
| C6a | 0.2253 (2) | 0.26206 (16) | 0.19085 (15) | 0.0279 (6) |
| H6aa | 0.218227 | 0.347891 | 0.227027 | 0.0335* |
| H6ab | 0.08712 | 0.206021 | 0.17726 | 0.0335* |
| H2b | −0.339134 | 0.554144 | 0.46148 | 0.075 (9)* |
| H3aa | 0.329781 | 0.119532 | 0.260701 | 0.045 (4)* |
| H3ab | 0.475827 | 0.248704 | 0.284554 | 0.045 (4)* |
| H3ac | 0.313145 | 0.21209 | 0.359189 | 0.045 (4)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1b | 0.0268 (6) | 0.0307 (7) | 0.0277 (6) | 0.0010 (5) | 0.0098 (5) | −0.0050 (5) |
| O2b | 0.0247 (6) | 0.0313 (7) | 0.0327 (7) | −0.0024 (5) | 0.0088 (5) | −0.0060 (5) |
| O3b | 0.0283 (6) | 0.0285 (7) | 0.0317 (7) | 0.0002 (5) | 0.0047 (5) | −0.0082 (5) |
| O4b | 0.0371 (7) | 0.0325 (7) | 0.0413 (8) | 0.0085 (6) | 0.0167 (6) | −0.0042 (6) |
| O5b | 0.0231 (6) | 0.0380 (8) | 0.0506 (9) | 0.0010 (6) | 0.0111 (6) | 0.0014 (6) |
| O6b | 0.0352 (7) | 0.0408 (8) | 0.0552 (10) | −0.0009 (6) | −0.0052 (7) | −0.0197 (7) |
| O7b | 0.0398 (7) | 0.0333 (7) | 0.0470 (8) | 0.0034 (6) | 0.0162 (6) | −0.0121 (6) |
| N1b | 0.0326 (8) | 0.0241 (8) | 0.0301 (8) | 0.0012 (6) | 0.0060 (6) | −0.0023 (6) |
| N2b | 0.0260 (7) | 0.0259 (7) | 0.0343 (8) | 0.0074 (6) | 0.0092 (6) | 0.0063 (6) |
| C1b | 0.0234 (8) | 0.0236 (8) | 0.0201 (8) | 0.0054 (6) | 0.0034 (6) | 0.0027 (6) |
| C2b | 0.0218 (8) | 0.0215 (8) | 0.0207 (8) | 0.0036 (6) | 0.0019 (6) | 0.0029 (6) |
| C3b | 0.0265 (8) | 0.0220 (8) | 0.0208 (8) | 0.0077 (7) | 0.0043 (6) | 0.0017 (6) |
| C4b | 0.0220 (8) | 0.0254 (8) | 0.0260 (8) | 0.0080 (7) | 0.0064 (7) | 0.0057 (7) |
| C5b | 0.0220 (8) | 0.0220 (8) | 0.0269 (8) | 0.0015 (6) | 0.0023 (7) | 0.0050 (7) |
| C6b | 0.0276 (8) | 0.0210 (8) | 0.0230 (8) | 0.0045 (7) | 0.0018 (7) | −0.0001 (6) |
| C7b | 0.0247 (8) | 0.0261 (8) | 0.0216 (8) | 0.0051 (7) | 0.0021 (6) | 0.0007 (6) |
| N1a | 0.0292 (8) | 0.0252 (8) | 0.0349 (8) | 0.0050 (6) | 0.0011 (6) | −0.0007 (6) |
| N2a | 0.0247 (7) | 0.0234 (7) | 0.0229 (7) | 0.0054 (6) | 0.0034 (5) | 0.0002 (6) |
| N3a | 0.0268 (7) | 0.0229 (7) | 0.0245 (7) | 0.0041 (6) | 0.0021 (6) | −0.0026 (6) |
| C1a | 0.0286 (9) | 0.0260 (9) | 0.0278 (9) | 0.0087 (7) | 0.0051 (7) | −0.0018 (7) |
| C2a | 0.0269 (9) | 0.0316 (10) | 0.0352 (10) | 0.0047 (7) | 0.0073 (7) | 0.0060 (8) |
| C3a | 0.0301 (9) | 0.0311 (9) | 0.0289 (9) | 0.0095 (7) | 0.0092 (7) | 0.0016 (7) |
| C4a | 0.0277 (8) | 0.0248 (8) | 0.0248 (8) | 0.0009 (7) | 0.0052 (7) | 0.0000 (7) |
| C5a | 0.0237 (8) | 0.0295 (9) | 0.0260 (9) | 0.0030 (7) | 0.0055 (7) | −0.0021 (7) |
| C6a | 0.0301 (9) | 0.0306 (9) | 0.0261 (9) | 0.0125 (7) | 0.0053 (7) | 0.0016 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1b—C1b | 1.284 (2) | N1a—C2a | 1.376 (3) |
| O2b—C7b | 1.308 (2) | N2a—C1a | 1.348 (2) |
| O2b—H2b | 0.9820 (12) | N2a—C3a | 1.375 (2) |
| O3b—C7b | 1.222 (2) | N2a—C4a | 1.4622 (19) |
| O4b—N2b | 1.232 (2) | N3a—C6a | 1.483 (2) |
| O5b—N2b | 1.2248 (17) | N3a—H3aa | 0.9042 (14) |

| | | | |
|---|---|---|---|
| O6b—N1b | 1.2313 (18) | N3a—H3ab | 0.9009 (13) |
| O7b—N1b | 1.225 (2) | N3a—H3ac | 0.8932 (14) |
| N1b—C6b | 1.464 (2) | C1a—H1a | 0.95 |
| N2b—C4b | 1.458 (2) | C2a—H2a | 0.95 |
| C1b—C2b | 1.440 (2) | C2a—C3a | 1.351 (2) |
| C1b—C6b | 1.428 (2) | C3a—H3a | 0.95 |
| C2b—C3b | 1.373 (2) | C4a—H4aa | 0.99 |
| C2b—C7b | 1.496 (2) | C4a—H4ab | 0.99 |
| C3b—H3b | 0.95 | C4a—C5a | 1.517 (2) |
| C3b—C4b | 1.382 (2) | C5a—H5aa | 0.99 |
| C4b—C5b | 1.378 (2) | C5a—H5ab | 0.99 |
| C5b—H5b | 0.95 | C5a—C6a | 1.507 (2) |
| C5b—C6b | 1.378 (2) | C6a—H6aa | 0.99 |
| N1a—C1a | 1.318 (2) | C6a—H6ab | 0.99 |

| | | | |
|---|---|---|---|
| C1b—O1b—H2b | 99.82 (10) | C6a—N3a—H3ab | 109.46 (13) |
| C7b—O2b—H2b | 106.90 (11) | C6a—N3a—H3ac | 111.76 (15) |
| O6b—N1b—O7b | 123.30 (15) | H3aa—N3a—H3ab | 110.21 (16) |
| O6b—N1b—C6b | 117.72 (16) | H3aa—N3a—H3ac | 106.71 (14) |
| O7b—N1b—C6b | 118.97 (13) | H3ab—N3a—H3ac | 107.27 (13) |
| O4b—N2b—O5b | 123.59 (16) | N1a—C1a—N2a | 111.91 (16) |
| O4b—N2b—C4b | 117.89 (12) | N1a—C1a—H1a | 124.04 |
| O5b—N2b—C4b | 118.52 (14) | N2a—C1a—H1a | 124.04 |
| O1b—C1b—C2b | 120.19 (13) | N1a—C2a—H2a | 124.86 |
| O1b—C1b—C6b | 124.76 (15) | N1a—C2a—C3a | 110.28 (15) |
| C2b—C1b—C6b | 115.00 (15) | H2a—C2a—C3a | 124.86 |
| C1b—C2b—C3b | 121.54 (13) | N2a—C3a—C2a | 106.10 (16) |
| C1b—C2b—C7b | 119.88 (15) | N2a—C3a—H3a | 126.95 |
| C3b—C2b—C7b | 118.56 (14) | C2a—C3a—H3a | 126.95 |
| C2b—C3b—H3b | 120.01 | N2a—C4a—H4aa | 109.47 |
| C2b—C3b—C4b | 119.99 (15) | N2a—C4a—H4ab | 109.47 |
| H3b—C3b—C4b | 120.01 | N2a—C4a—C5a | 112.62 (14) |
| N2b—C4b—C3b | 119.03 (14) | H4aa—C4a—H4ab | 106.12 |
| N2b—C4b—C5b | 119.24 (13) | H4aa—C4a—C5a | 109.47 |
| C3b—C4b—C5b | 121.73 (16) | H4ab—C4a—C5a | 109.47 |
| C4b—C5b—H5b | 120.69 | C4a—C5a—H5aa | 109.47 |
| C4b—C5b—C6b | 118.62 (13) | C4a—C5a—H5ab | 109.47 |
| H5b—C5b—C6b | 120.69 | C4a—C5a—C6a | 111.50 (15) |
| N1b—C6b—C1b | 120.26 (15) | H5aa—C5a—H5ab | 107.36 |
| N1b—C6b—C5b | 116.59 (13) | H5aa—C5a—C6a | 109.47 |
| C1b—C6b—C5b | 123.09 (15) | H5ab—C5a—C6a | 109.47 |
| O2b—C7b—O3b | 121.86 (14) | N3a—C6a—C5a | 112.35 (15) |
| O2b—C7b—C2b | 115.78 (14) | N3a—C6a—H6aa | 109.47 |
| O3b—C7b—C2b | 122.33 (16) | N3a—C6a—H6ab | 109.47 |
| C1a—N1a—C2a | 105.01 (14) | C5a—C6a—H6aa | 109.47 |
| C1a—N2a—C3a | 106.69 (13) | C5a—C6a—H6ab | 109.47 |
| C1a—N2a—C4a | 125.73 (15) | H6aa—C6a—H6ab | 106.43 |
| C3a—N2a—C4a | 127.56 (14) | O1b—H2b—O2b | 156.29 (9) |

| | |
|---|---|
| C6a—N3a—H3aa | 111.33 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4*a*—H4*aa*···O4*b*$^{i}$ | 0.99 | 2.53 | 3.359 (2) | 141 |
| O2*b*—H2*b*···O1*b* | 0.9820 (12) | 1.5161 (11) | 2.4473 (16) | 156.29 (9) |
| O2*b*—H2*b*···C1*b* | 0.9820 (12) | 2.1471 (15) | 2.7833 (18) | 121.00 (8) |
| N3*a*—H3*aa*···N1*a*$^{ii}$ | 0.9042 (14) | 1.9318 (14) | 2.797 (2) | 159.6 (1) |
| N3*a*—H3*ab*···O2*b*$^{iii}$ | 0.9009 (13) | 2.5650 (12) | 3.1297 (17) | 121.35 (10) |
| N3*a*—H3*ab*···O3*b*$^{iii}$ | 0.9009 (13) | 2.0721 (11) | 2.9537 (17) | 165.79 (10) |
| N3*a*—H3*ac*···O1*b*$^{iv}$ | 0.8932 (14) | 2.0610 (13) | 2.815 (2) | 141.47 (11) |
| N3*a*—H3*ac*···O7*b*$^{iv}$ | 0.8932 (14) | 2.4844 (13) | 2.9712 (19) | 114.74 (8) |

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

## 4-{[(5-Methylisoxazol-3-yl)amino]sulfonyl}anilinium 2-hydroxy-3,5-dinitrobenzoate (TUJPEV)

*Crystal data*

$C_{10}H_{12}N_3O_3S^+·C_7H_3N_2O_7^-$
$M_r$ = 481.41
Triclinic, $P\overline{1}$
Hall symbol: -P 1
*a* = 8.5551 (1) Å
*b* = 10.5000 (2) Å
*c* = 12.7576 (3) Å
*α* = 106.463 (1)°
*β* = 100.913 (1)°
*γ* = 108.272 (1)°
*V* = 993.72 (3) Å$^3$
*Z* = 2
*F*(000) = 496
$D_x$ = 1.609 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6718 reflections
*θ* = 1.8–32.6°
*μ* = 0.23 mm$^{-1}$
*T* = 296 K
Prism, yellow
0.20 × 0.20 × 0.16 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*ω* and *φ* scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
$T_{min}$ = 0.955, $T_{max}$ = 0.964
24261 measured reflections
6717 independent reflections
4398 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.030
$\theta_{max}$ = 32.6°, $\theta_{min}$ = 1.8°
*h* = −12→12
*k* = −15→15
*l* = −19→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)]$ = 0.044
$wR(F)$ = 0.104
*S* = 1.95
6717 reflections
301 parameters
0 restraints
48 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 0.31 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.
Number of fixed parameters: 9

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | −0.02012 (4) | 0.65156 (4) | 0.38514 (3) | 0.03475 (15) |
| O1 | −0.12136 (12) | 0.56298 (10) | 0.26952 (9) | 0.0472 (5) |
| O2 | −0.09555 (13) | 0.65440 (11) | 0.47594 (9) | 0.0468 (5) |
| O3 | 0.47434 (14) | 0.55510 (13) | 0.32058 (11) | 0.0626 (6) |
| N1 | 0.29155 (15) | 1.24477 (12) | 0.38430 (11) | 0.0434 (6) |
| N2 | 0.13845 (14) | 0.60248 (12) | 0.42019 (10) | 0.0376 (5) |
| N3 | 0.39246 (16) | 0.57705 (15) | 0.40555 (12) | 0.0536 (6) |
| C1 | 0.07464 (15) | 0.82919 (13) | 0.38935 (12) | 0.0322 (5) |
| C2 | 0.21693 (19) | 0.93183 (16) | 0.47844 (13) | 0.0511 (7) |
| C3 | 0.28732 (19) | 1.06857 (16) | 0.47796 (14) | 0.0514 (7) |
| C4 | 0.21467 (16) | 1.10142 (13) | 0.38945 (12) | 0.0344 (6) |
| C5 | 0.07052 (18) | 1.00124 (15) | 0.30229 (13) | 0.0444 (7) |
| C6 | 0.00046 (17) | 0.86380 (15) | 0.30181 (13) | 0.0421 (6) |
| C7 | 0.24788 (17) | 0.58299 (13) | 0.35471 (13) | 0.0355 (6) |
| C8 | 0.2306 (2) | 0.56568 (16) | 0.23936 (14) | 0.0473 (7) |
| C9 | 0.3761 (2) | 0.55015 (16) | 0.22381 (16) | 0.0511 (8) |
| C10 | 0.4452 (3) | 0.5287 (2) | 0.12416 (18) | 0.0766 (11) |
| O4 | 0.51685 (14) | 0.76882 (14) | 0.75724 (10) | 0.0657 (6) |
| O5 | 0.25004 (13) | 0.72390 (11) | 0.65907 (9) | 0.0495 (5) |
| O6 | 0.03442 (11) | 0.74382 (11) | 0.75483 (8) | 0.0431 (4) |
| O7 | −0.13539 (17) | 0.8490 (2) | 0.89544 (14) | 0.0980 (10) |
| O8 | −0.12065 (16) | 0.7660 (2) | 1.03000 (12) | 0.0975 (9) |
| O9 | 0.46550 (16) | 0.90412 (15) | 1.25865 (10) | 0.0717 (7) |
| O10 | 0.66293 (15) | 0.90569 (15) | 1.17611 (11) | 0.0749 (7) |
| N4 | −0.06158 (16) | 0.80710 (17) | 0.96062 (13) | 0.0598 (7) |
| N5 | 0.51466 (16) | 0.88865 (13) | 1.17407 (12) | 0.0488 (6) |
| C11 | 0.31789 (15) | 0.78551 (13) | 0.85956 (12) | 0.0326 (5) |
| C12 | 0.14714 (16) | 0.77857 (13) | 0.85292 (12) | 0.0328 (5) |
| C13 | 0.10840 (16) | 0.80786 (15) | 0.95755 (13) | 0.0389 (6) |
| C14 | 0.22578 (17) | 0.84024 (15) | 1.06095 (13) | 0.0403 (6) |
| C15 | 0.38931 (16) | 0.84763 (14) | 1.06263 (12) | 0.0365 (6) |
| C16 | 0.43735 (16) | 0.82163 (13) | 0.96389 (12) | 0.0356 (6) |
| C17 | 0.36856 (18) | 0.75728 (15) | 0.75210 (13) | 0.0400 (6) |
| H1a | 0.188848 | 1.256572 | 0.342213 | 0.088 (4)* |

| | | | | |
|---|---|---|---|---|
| H1b | 0.353568 | 1.297412 | 0.446823 | 0.088 (4)* |
| H1c | 0.348288 | 1.238627 | 0.338232 | 0.088 (4)* |
| H2 | 0.265121 | 0.908789 | 0.538567 | 0.0613* |
| H2a | 0.186499 | 0.630039 | 0.50234 | 0.068 (5)* |
| H3 | 0.383907 | 1.138439 | 0.537561 | 0.0617* |
| H5 | 0.020222 | 1.025766 | 0.243815 | 0.0533* |
| H6 | −0.096798 | 0.794538 | 0.242417 | 0.0506* |
| H8 | 0.139615 | 0.565054 | 0.185878 | 0.0568* |
| H10a | 0.551947 | 0.608597 | 0.142286 | 0.1149* |
| H10b | 0.362955 | 0.522551 | 0.058321 | 0.1149* |
| H10c | 0.46505 | 0.441064 | 0.1077 | 0.1149* |
| H14 | 0.194965 | 0.856742 | 1.128171 | 0.0484* |
| H16 | 0.549353 | 0.82833 | 0.967386 | 0.0428* |
| H6a | 0.115577 | 0.727699 | 0.69023 | 0.132 (9)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.03683 (17) | 0.03321 (18) | 0.0360 (2) | 0.01370 (13) | 0.01260 (14) | 0.01423 (16) |
| O1 | 0.0462 (5) | 0.0373 (5) | 0.0422 (7) | 0.0089 (4) | 0.0009 (5) | 0.0087 (5) |
| O2 | 0.0524 (6) | 0.0510 (6) | 0.0532 (7) | 0.0246 (5) | 0.0309 (5) | 0.0276 (6) |
| O3 | 0.0518 (6) | 0.0718 (8) | 0.0660 (9) | 0.0313 (6) | 0.0248 (6) | 0.0153 (7) |
| N1 | 0.0510 (7) | 0.0374 (6) | 0.0502 (9) | 0.0178 (5) | 0.0268 (6) | 0.0202 (6) |
| N2 | 0.0476 (6) | 0.0413 (6) | 0.0327 (7) | 0.0235 (5) | 0.0154 (5) | 0.0169 (6) |
| N3 | 0.0495 (7) | 0.0628 (9) | 0.0487 (9) | 0.0283 (6) | 0.0154 (6) | 0.0136 (7) |
| C1 | 0.0350 (6) | 0.0317 (7) | 0.0316 (8) | 0.0143 (5) | 0.0112 (5) | 0.0120 (6) |
| C2 | 0.0588 (9) | 0.0433 (9) | 0.0382 (9) | 0.0107 (7) | −0.0053 (7) | 0.0197 (8) |
| C3 | 0.0520 (9) | 0.0380 (8) | 0.0432 (10) | 0.0029 (7) | −0.0033 (7) | 0.0129 (8) |
| C4 | 0.0400 (7) | 0.0320 (7) | 0.0393 (9) | 0.0172 (6) | 0.0208 (6) | 0.0153 (6) |
| C5 | 0.0478 (8) | 0.0446 (8) | 0.0438 (10) | 0.0195 (7) | 0.0064 (7) | 0.0235 (8) |
| C6 | 0.0392 (7) | 0.0397 (8) | 0.0403 (9) | 0.0118 (6) | 0.0004 (6) | 0.0159 (7) |
| C7 | 0.0414 (7) | 0.0275 (7) | 0.0374 (9) | 0.0132 (5) | 0.0131 (6) | 0.0113 (6) |
| C8 | 0.0553 (9) | 0.0506 (9) | 0.0426 (10) | 0.0238 (7) | 0.0203 (7) | 0.0194 (8) |
| C9 | 0.0605 (9) | 0.0379 (8) | 0.0574 (12) | 0.0169 (7) | 0.0325 (9) | 0.0141 (8) |
| C10 | 0.0921 (14) | 0.0731 (13) | 0.0819 (15) | 0.0351 (12) | 0.0607 (13) | 0.0275 (13) |
| O4 | 0.0483 (6) | 0.0981 (10) | 0.0513 (8) | 0.0274 (6) | 0.0254 (5) | 0.0227 (7) |
| O5 | 0.0578 (6) | 0.0613 (7) | 0.0295 (6) | 0.0241 (5) | 0.0132 (5) | 0.0156 (5) |
| O6 | 0.0404 (5) | 0.0525 (6) | 0.0322 (6) | 0.0196 (4) | 0.0028 (4) | 0.0136 (5) |
| O7 | 0.0700 (8) | 0.1711 (16) | 0.0831 (11) | 0.0810 (10) | 0.0212 (8) | 0.0529 (11) |
| O8 | 0.0537 (7) | 0.1721 (16) | 0.0566 (9) | 0.0299 (9) | 0.0287 (7) | 0.0359 (10) |
| O9 | 0.0770 (8) | 0.0950 (10) | 0.0308 (7) | 0.0267 (7) | 0.0041 (6) | 0.0211 (7) |
| O10 | 0.0480 (7) | 0.0991 (10) | 0.0639 (9) | 0.0302 (6) | −0.0064 (6) | 0.0241 (8) |
| N4 | 0.0396 (7) | 0.0856 (11) | 0.0419 (9) | 0.0249 (7) | 0.0082 (6) | 0.0084 (8) |
| N5 | 0.0478 (7) | 0.0458 (7) | 0.0405 (9) | 0.0147 (6) | −0.0044 (6) | 0.0138 (7) |
| C11 | 0.0346 (6) | 0.0281 (6) | 0.0318 (8) | 0.0107 (5) | 0.0078 (5) | 0.0096 (6) |
| C12 | 0.0351 (6) | 0.0301 (7) | 0.0291 (8) | 0.0112 (5) | 0.0039 (5) | 0.0104 (6) |
| C13 | 0.0330 (6) | 0.0444 (8) | 0.0356 (9) | 0.0154 (6) | 0.0079 (6) | 0.0108 (7) |
| C14 | 0.0424 (7) | 0.0454 (8) | 0.0301 (8) | 0.0162 (6) | 0.0100 (6) | 0.0112 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.0370 (7) | 0.0341 (7) | 0.0297 (8) | 0.0107 (5) | −0.0009 (6) | 0.0104 (6) |
| C16 | 0.0321 (6) | 0.0324 (7) | 0.0393 (9) | 0.0124 (5) | 0.0061 (6) | 0.0120 (6) |
| C17 | 0.0411 (7) | 0.0393 (8) | 0.0385 (9) | 0.0139 (6) | 0.0130 (6) | 0.0143 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O1 | 1.4228 (9) | C9—C10 | 1.491 (3) |
| S1—O2 | 1.4264 (13) | C10—H10a | 0.96 |
| S1—N2 | 1.6264 (14) | C10—H10b | 0.96 |
| O3—N3 | 1.402 (2) | C10—H10c | 0.96 |
| O3—C9 | 1.333 (2) | O4—C17 | 1.223 (2) |
| N1—C4 | 1.468 (2) | O5—C17 | 1.2827 (18) |
| N1—H1a | 1.0015 (14) | O5—H6a | 1.2945 (12) |
| N1—H1b | 0.7932 (11) | O6—C12 | 1.3010 (16) |
| N1—H1c | 0.8315 (15) | O6—H6a | 1.1837 (11) |
| N2—C7 | 1.391 (2) | O7—N4 | 1.211 (3) |
| N2—H2a | 0.9703 (12) | O8—N4 | 1.214 (2) |
| N3—C7 | 1.312 (2) | O9—N5 | 1.218 (2) |
| C1—C2 | 1.3764 (15) | O10—N5 | 1.218 (2) |
| C1—C6 | 1.374 (2) | N4—C13 | 1.460 (2) |
| C2—C3 | 1.374 (2) | N5—C15 | 1.4638 (19) |
| C2—H2 | 0.93 | C11—C12 | 1.425 (2) |
| C3—C4 | 1.367 (2) | C11—C16 | 1.3841 (19) |
| C3—H3 | 0.93 | C11—C17 | 1.493 (2) |
| C4—C5 | 1.3668 (15) | C12—C13 | 1.409 (2) |
| C5—C6 | 1.376 (2) | C13—C14 | 1.376 (2) |
| C5—H5 | 0.93 | C14—C15 | 1.372 (2) |
| C6—H6 | 0.93 | C14—H14 | 0.93 |
| C7—C8 | 1.403 (2) | C15—C16 | 1.379 (2) |
| C8—C9 | 1.348 (3) | C16—H16 | 0.93 |
| C8—H8 | 0.93 | | |

| | | | |
|---|---|---|---|
| O1—S1—O2 | 120.50 (6) | C8—C9—C10 | 133.88 (19) |
| O1—S1—N2 | 108.78 (6) | C9—C10—H10a | 109.47 |
| O2—S1—N2 | 104.15 (7) | C9—C10—H10b | 109.47 |
| N3—O3—C9 | 108.94 (14) | C9—C10—H10c | 109.47 |
| C4—N1—H1a | 102.59 (10) | H10a—C10—H10b | 109.47 |
| C4—N1—H1b | 107.06 (15) | H10a—C10—H10c | 109.47 |
| C4—N1—H1c | 107.83 (13) | H10b—C10—H10c | 109.47 |
| H1a—N1—H1b | 124.65 (16) | C17—O5—H6a | 105.28 (11) |
| H1a—N1—H1c | 103.41 (14) | C12—O6—H6a | 102.01 (10) |
| H1b—N1—H1c | 110.21 (14) | O7—N4—O8 | 123.34 (17) |
| S1—N2—C7 | 124.43 (12) | O7—N4—C13 | 118.74 (17) |
| S1—N2—H2a | 113.65 (11) | O8—N4—C13 | 117.91 (17) |
| C7—N2—H2a | 116.39 (12) | O9—N5—O10 | 124.03 (14) |
| O3—N3—C7 | 104.86 (14) | O9—N5—C15 | 118.56 (14) |
| C2—C1—C6 | 120.38 (14) | O10—N5—C15 | 117.41 (15) |
| C1—C2—C3 | 119.57 (16) | C12—C11—C16 | 120.99 (14) |

| | | | |
|---|---|---|---|
| C1—C2—H2 | 120.22 | C12—C11—C17 | 118.93 (12) |
| C3—C2—H2 | 120.22 | C16—C11—C17 | 120.07 (13) |
| C2—C3—C4 | 119.68 (12) | O6—C12—C11 | 121.05 (14) |
| C2—C3—H3 | 120.16 | O6—C12—C13 | 122.90 (13) |
| C4—C3—H3 | 120.16 | C11—C12—C13 | 116.03 (12) |
| N1—C4—C3 | 120.80 (10) | N4—C13—C12 | 120.40 (13) |
| N1—C4—C5 | 118.09 (15) | N4—C13—C14 | 116.57 (15) |
| C3—C4—C5 | 121.09 (14) | C12—C13—C14 | 123.02 (14) |
| C4—C5—C6 | 119.44 (16) | C13—C14—C15 | 118.50 (15) |
| C4—C5—H5 | 120.28 | C13—C14—H14 | 120.75 |
| C6—C5—H5 | 120.28 | C15—C14—H14 | 120.75 |
| C1—C6—C5 | 119.80 (11) | N5—C15—C14 | 117.72 (14) |
| C1—C6—H6 | 120.1 | N5—C15—C16 | 120.43 (13) |
| C5—C6—H6 | 120.1 | C14—C15—C16 | 121.82 (13) |
| N2—C7—N3 | 117.05 (14) | C11—C16—C15 | 119.61 (13) |
| N2—C7—C8 | 131.01 (14) | C11—C16—H16 | 120.19 |
| N3—C7—C8 | 111.93 (15) | C15—C16—H16 | 120.19 |
| C7—C8—C9 | 104.20 (16) | O4—C17—O5 | 124.29 (16) |
| C7—C8—H8 | 127.9 | O4—C17—C11 | 119.59 (13) |
| C9—C8—H8 | 127.9 | O5—C17—C11 | 116.11 (13) |
| O3—C9—C8 | 110.05 (18) | O5—H6a—O6 | 156.58 (6) |
| O3—C9—C10 | 116.07 (17) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*a*···O6$^{i}$ | 1.0015 (14) | 2.0683 (10) | 3.0655 (17) | 173.55 (7) |
| N1—H1*b*···N3$^{ii}$ | 0.7932 (11) | 2.2920 (11) | 3.0393 (15) | 157.33 (11) |
| N1—H1*c*···O4$^{ii}$ | 0.8315 (15) | 1.8318 (14) | 2.663 (2) | 177.12 (7) |
| N2—H2*a*···O5 | 0.9703 (12) | 1.8440 (10) | 2.7852 (15) | 162.64 (9) |
| O5—H6*a*···O6 | 1.2945 (12) | 1.1837 (11) | 2.4268 (16) | 156.58 (6) |
| O5—H6*a*···C12 | 1.2945 (12) | 1.9326 (15) | 2.7490 (19) | 115.40 (6) |
| O6—H6*a*···O5 | 1.1837 (11) | 1.2945 (12) | 2.4268 (16) | 156.58 (6) |
| O6—H6*a*···C17 | 1.1837 (11) | 2.0485 (15) | 2.8249 (19) | 119.42 (7) |

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

## 2-Isopropyl-6-methyl-4-oxo-3,4-dihydropyrimidin-1-ium 2-carboxy-4,6-dinitrophenolate monohydrate (VABZIJ)

*Crystal data*

$C_8H_{13}N_2O^+·C_7H_3N_2O_7^-·H_2O$
$M_r$ = 398.33
Triclinic, $P\overline{1}$
Hall symbol: -P 1
*a* = 6.6691 (3) Å
*b* = 11.3831 (4) Å
*c* = 12.2900 (5) Å
*α* = 89.727 (2)°
*β* = 76.771 (2)°
*γ* = 76.930 (2)°
*V* = 883.62 (6) Å$^3$
*Z* = 2
*F*(000) = 416
$D_x$ = 1.497 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6994 reflections
*θ* = 2.4–31.6°
*μ* = 0.13 mm$^{-1}$
*T* = 100 K
Block, yellow
0.52 × 0.13 × 0.10 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
$T_{min}$ = 0.937, $T_{max}$ = 0.987
17014 measured reflections
4061 independent reflections
3042 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.030
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.7°
$h$ = −8→8
$k$ = −14→12
$l$ = −15→15

*Refinement*

Refinement on $F^2$
$R[F > 3\sigma(F)]$ = 0.038
$wR(F)$ = 0.086
$S$ = 1.77
4061 reflections
258 parameters
0 restraints
52 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max}$ = 0.014
$\Delta\rho_{max}$ = 0.46 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. Number of fixed parameters: 15

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.30546 (16) | −0.17099 (9) | 0.14963 (8) | 0.0232 (4) |
| O2 | 0.56653 (17) | −0.35718 (9) | 0.01361 (9) | 0.0316 (4) |
| O3 | 0.48656 (18) | −0.36691 (9) | −0.14697 (9) | 0.0311 (4) |
| O4 | 0.36254 (16) | −0.00313 (9) | −0.33889 (8) | 0.0235 (4) |
| O5 | 0.23753 (16) | 0.16995 (9) | −0.24573 (8) | 0.0255 (4) |
| O6 | 0.07250 (15) | 0.19153 (8) | 0.16330 (8) | 0.0204 (4) |
| O7 | 0.14533 (16) | 0.02234 (9) | 0.25238 (8) | 0.0215 (4) |
| O8 | 0.31889 (16) | 0.38510 (9) | 0.41956 (8) | 0.0221 (4) |
| N1 | 0.30174 (18) | 0.05930 (11) | −0.25094 (10) | 0.0185 (4) |
| N2 | 0.48606 (19) | −0.31064 (11) | −0.06125 (10) | 0.0213 (4) |
| N3 | 0.11898 (17) | 0.64252 (10) | 0.66449 (9) | 0.0147 (4) |
| N4 | 0.23841 (17) | 0.58259 (10) | 0.47936 (9) | 0.0144 (4) |
| C1 | 0.3103 (2) | −0.11910 (12) | 0.05547 (11) | 0.0151 (5) |
| C2 | 0.3895 (2) | −0.18160 (12) | −0.05145 (11) | 0.0154 (5) |

| | | | | |
|---|---|---|---|---|
| C3 | 0.3838 (2) | −0.12419 (13) | −0.15034 (12) | 0.0160 (5) |
| H3a | 0.432743 | −0.167918 | −0.218686 | 0.0192* |
| C4 | 0.3044 (2) | −0.00126 (13) | −0.14615 (11) | 0.0150 (5) |
| C5 | 0.2281 (2) | 0.06626 (13) | −0.04539 (11) | 0.0148 (5) |
| H5a | 0.176964 | 0.149404 | −0.044656 | 0.0177* |
| C6 | 0.2290 (2) | 0.00871 (12) | 0.05379 (11) | 0.0140 (5) |
| C7 | 0.1429 (2) | 0.08121 (13) | 0.16123 (11) | 0.0159 (5) |
| C8 | 0.2598 (2) | 0.45829 (12) | 0.49910 (12) | 0.0158 (5) |
| C9 | 0.2070 (2) | 0.43367 (13) | 0.61582 (11) | 0.0160 (5) |
| H9a | 0.221191 | 0.354014 | 0.636387 | 0.0192* |
| C10 | 0.1376 (2) | 0.52320 (12) | 0.69587 (11) | 0.0157 (5) |
| C11 | 0.1694 (2) | 0.67095 (12) | 0.55875 (11) | 0.0141 (5) |
| C12 | 0.1445 (2) | 0.80006 (12) | 0.52928 (11) | 0.0153 (5) |
| H12a | 0.110348 | 0.85057 | 0.598371 | 0.0184* |
| C13 | −0.0374 (2) | 0.83589 (13) | 0.47019 (13) | 0.0224 (5) |
| H13a | −0.054927 | 0.919318 | 0.452627 | 0.0335* |
| H13b | −0.165759 | 0.824046 | 0.518462 | 0.0335* |
| H13c | −0.005734 | 0.786763 | 0.402409 | 0.0335* |
| C14 | 0.3509 (2) | 0.82298 (12) | 0.45745 (12) | 0.0195 (5) |
| H14a | 0.461527 | 0.800548 | 0.496897 | 0.0292* |
| H14b | 0.331515 | 0.907081 | 0.442205 | 0.0292* |
| H14c | 0.389104 | 0.775688 | 0.38823 | 0.0292* |
| C15 | 0.0772 (2) | 0.50600 (13) | 0.81821 (12) | 0.0221 (5) |
| H15a | −0.068894 | 0.545223 | 0.847126 | 0.0332* |
| H15b | 0.163895 | 0.540389 | 0.855439 | 0.0332* |
| H15c | 0.097793 | 0.421295 | 0.830953 | 0.0332* |
| O1w | 0.32852 (17) | 0.60955 (9) | 0.25340 (8) | 0.0272 (4) |
| H1n3 | 0.061415 | 0.706878 | 0.722609 | 0.037 (5)* |
| H1n4 | 0.270467 | 0.602234 | 0.406704 | 0.034 (5)* |
| H2w1 | 0.339729 | 0.675124 | 0.210779 | 0.053 (6)* |
| H1w1 | 0.381611 | 0.539911 | 0.20932 | 0.055 (6)* |
| H7 | 0.211364 | −0.063754 | 0.220331 | 0.078 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0305 (6) | 0.0178 (6) | 0.0185 (6) | −0.0038 (5) | −0.0018 (5) | 0.0031 (4) |
| O2 | 0.0431 (7) | 0.0219 (6) | 0.0232 (6) | 0.0045 (5) | −0.0063 (5) | 0.0044 (5) |
| O3 | 0.0489 (7) | 0.0181 (6) | 0.0229 (6) | −0.0036 (5) | −0.0053 (5) | −0.0086 (5) |
| O4 | 0.0291 (6) | 0.0274 (6) | 0.0138 (6) | −0.0083 (5) | −0.0030 (5) | −0.0009 (5) |
| O5 | 0.0347 (6) | 0.0178 (6) | 0.0235 (6) | −0.0045 (5) | −0.0072 (5) | 0.0058 (5) |
| O6 | 0.0244 (6) | 0.0142 (6) | 0.0187 (6) | −0.0013 (4) | −0.0004 (4) | −0.0029 (4) |
| O7 | 0.0284 (6) | 0.0175 (6) | 0.0140 (5) | −0.0009 (5) | 0.0003 (4) | −0.0003 (4) |
| O8 | 0.0321 (6) | 0.0137 (5) | 0.0176 (6) | −0.0026 (5) | −0.0027 (5) | −0.0025 (4) |
| N1 | 0.0182 (6) | 0.0202 (7) | 0.0186 (7) | −0.0071 (6) | −0.0047 (5) | 0.0034 (6) |
| N2 | 0.0240 (7) | 0.0161 (7) | 0.0198 (7) | −0.0035 (6) | 0.0019 (6) | 0.0001 (6) |
| N3 | 0.0168 (6) | 0.0123 (6) | 0.0136 (6) | −0.0024 (5) | −0.0017 (5) | −0.0006 (5) |
| N4 | 0.0172 (6) | 0.0117 (6) | 0.0129 (6) | −0.0026 (5) | −0.0014 (5) | 0.0011 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.0133 (7) | 0.0177 (8) | 0.0148 (7) | −0.0064 (6) | −0.0014 (6) | 0.0022 (6) |
| C2 | 0.0158 (7) | 0.0118 (7) | 0.0176 (8) | −0.0030 (6) | −0.0017 (6) | −0.0008 (6) |
| C3 | 0.0159 (7) | 0.0184 (8) | 0.0140 (8) | −0.0070 (6) | −0.0012 (6) | −0.0029 (6) |
| C4 | 0.0146 (7) | 0.0196 (8) | 0.0125 (7) | −0.0073 (6) | −0.0030 (6) | 0.0033 (6) |
| C5 | 0.0121 (7) | 0.0137 (7) | 0.0188 (8) | −0.0046 (6) | −0.0026 (6) | 0.0007 (6) |
| C6 | 0.0113 (7) | 0.0154 (7) | 0.0154 (7) | −0.0048 (6) | −0.0015 (6) | −0.0005 (6) |
| C7 | 0.0130 (7) | 0.0171 (8) | 0.0170 (8) | −0.0048 (6) | −0.0008 (6) | 0.0000 (6) |
| C8 | 0.0145 (7) | 0.0132 (7) | 0.0193 (8) | −0.0022 (6) | −0.0036 (6) | 0.0001 (6) |
| C9 | 0.0177 (7) | 0.0110 (7) | 0.0187 (8) | −0.0031 (6) | −0.0036 (6) | 0.0032 (6) |
| C10 | 0.0137 (7) | 0.0154 (8) | 0.0182 (8) | −0.0033 (6) | −0.0041 (6) | 0.0029 (6) |
| C11 | 0.0106 (7) | 0.0152 (7) | 0.0161 (8) | −0.0028 (6) | −0.0026 (6) | −0.0012 (6) |
| C12 | 0.0178 (7) | 0.0114 (7) | 0.0155 (7) | −0.0034 (6) | −0.0014 (6) | −0.0007 (6) |
| C13 | 0.0218 (8) | 0.0140 (8) | 0.0321 (9) | −0.0033 (6) | −0.0089 (7) | 0.0052 (7) |
| C14 | 0.0210 (8) | 0.0141 (8) | 0.0218 (8) | −0.0048 (6) | −0.0012 (6) | 0.0004 (6) |
| C15 | 0.0269 (8) | 0.0194 (8) | 0.0188 (8) | −0.0047 (7) | −0.0034 (6) | 0.0019 (6) |
| O1w | 0.0471 (7) | 0.0134 (6) | 0.0163 (6) | −0.0043 (5) | −0.0005 (5) | 0.0003 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.2939 (17) | C4—C5 | 1.3881 (18) |
| O1—H7 | 1.4329 (9) | C5—H5a | 0.93 |
| O2—N2 | 1.2279 (17) | C5—C6 | 1.3816 (19) |
| O3—N2 | 1.2346 (17) | C6—C7 | 1.4844 (18) |
| O4—N1 | 1.2304 (15) | C8—C9 | 1.439 (2) |
| O5—N1 | 1.2321 (15) | C9—H9a | 0.93 |
| O6—C7 | 1.2355 (16) | C9—C10 | 1.3449 (19) |
| O7—C7 | 1.3040 (17) | C10—C15 | 1.4889 (19) |
| O7—H7 | 1.0191 (9) | C11—C12 | 1.4936 (19) |
| O8—C8 | 1.2198 (17) | C12—H12a | 0.98 |
| N1—C4 | 1.4597 (18) | C12—C13 | 1.531 (2) |
| N2—C2 | 1.4576 (17) | C12—C14 | 1.5318 (19) |
| N3—C10 | 1.3963 (18) | C13—H13a | 0.96 |
| N3—C11 | 1.3236 (17) | C13—H13b | 0.96 |
| N3—H1n3 | 0.9729 (11) | C13—H13c | 0.96 |
| N4—C8 | 1.4152 (18) | C14—H14a | 0.96 |
| N4—C11 | 1.3303 (17) | C14—H14b | 0.96 |
| N4—H1n4 | 0.9085 (11) | C14—H14c | 0.96 |
| C1—C2 | 1.4259 (18) | C15—H15a | 0.96 |
| C1—C6 | 1.4349 (19) | C15—H15b | 0.96 |
| C2—C3 | 1.381 (2) | C15—H15c | 0.96 |
| C3—H3a | 0.93 | O1w—H2w1 | 0.9169 (10) |
| C3—C4 | 1.3763 (19) | O1w—H1w1 | 0.9146 (10) |
| | | | |
| C1—O1—H7 | 96.54 (8) | N4—C8—C9 | 113.60 (12) |
| C7—O7—H7 | 101.27 (9) | C8—C9—H9a | 119.24 |
| O4—N1—O5 | 124.04 (12) | C8—C9—C10 | 121.51 (13) |
| O4—N1—C4 | 118.12 (11) | H9a—C9—C10 | 119.24 |
| O5—N1—C4 | 117.84 (11) | N3—C10—C9 | 118.95 (12) |

| | | | |
|---|---|---|---|
| O2—N2—O3 | 123.42 (12) | N3—C10—C15 | 116.00 (11) |
| O2—N2—C2 | 119.16 (12) | C9—C10—C15 | 125.06 (13) |
| O3—N2—C2 | 117.39 (12) | N3—C11—N4 | 118.76 (12) |
| C10—N3—C11 | 122.45 (11) | N3—C11—C12 | 120.45 (12) |
| C10—N3—H1n3 | 118.37 (11) | N4—C11—C12 | 120.78 (12) |
| C11—N3—H1n3 | 119.14 (11) | C11—C12—H12a | 108.71 |
| C8—N4—C11 | 124.69 (12) | C11—C12—C13 | 109.61 (13) |
| C8—N4—H1n4 | 116.55 (11) | C11—C12—C14 | 111.42 (10) |
| C11—N4—H1n4 | 118.72 (12) | H12a—C12—C13 | 108.78 |
| O1—C1—C2 | 124.06 (12) | H12a—C12—C14 | 106.87 |
| O1—C1—C6 | 120.40 (11) | C13—C12—C14 | 111.36 (12) |
| C2—C1—C6 | 115.53 (12) | C12—C13—H13a | 109.47 |
| N2—C2—C1 | 120.85 (12) | C12—C13—H13b | 109.47 |
| N2—C2—C3 | 116.56 (11) | C12—C13—H13c | 109.47 |
| C1—C2—C3 | 122.58 (12) | H13a—C13—H13b | 109.47 |
| C2—C3—H3a | 120.52 | H13a—C13—H13c | 109.47 |
| C2—C3—C4 | 118.97 (12) | H13b—C13—H13c | 109.47 |
| H3a—C3—C4 | 120.52 | C12—C14—H14a | 109.47 |
| N1—C4—C3 | 118.77 (12) | C12—C14—H14b | 109.47 |
| N1—C4—C5 | 119.38 (12) | C12—C14—H14c | 109.47 |
| C3—C4—C5 | 121.85 (13) | H14a—C14—H14b | 109.47 |
| C4—C5—H5a | 120.33 | H14a—C14—H14c | 109.47 |
| C4—C5—C6 | 119.34 (12) | H14b—C14—H14c | 109.47 |
| H5a—C5—C6 | 120.33 | C10—C15—H15a | 109.47 |
| C1—C6—C5 | 121.71 (12) | C10—C15—H15b | 109.47 |
| C1—C6—C7 | 119.30 (12) | C10—C15—H15c | 109.47 |
| C5—C6—C7 | 118.99 (12) | H15a—C15—H15b | 109.47 |
| O6—C7—O7 | 122.20 (12) | H15a—C15—H15c | 109.47 |
| O6—C7—C6 | 121.26 (12) | H15b—C15—H15c | 109.47 |
| O7—C7—C6 | 116.54 (12) | H2w1—O1w—H1w1 | 110.00 (10) |
| O8—C8—N4 | 119.14 (12) | O1—H7—O7 | 165.94 (7) |
| O8—C8—C9 | 127.26 (13) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C12—H12*a*···O7$^{i}$ | 0.98 | 2.42 | 3.3068 (15) | 151 |
| N3—H1*n*3···O6$^{i}$ | 0.9729 (11) | 1.7539 (9) | 2.7182 (14) | 170.48 (8) |
| N4—H1*n*4···O1*w* | 0.9085 (11) | 1.8401 (10) | 2.7348 (15) | 167.76 (8) |
| O1*w*—H2*w*1···O1$^{ii}$ | 0.9169 (10) | 1.8895 (10) | 2.7886 (14) | 166.21 (6) |
| O1*w*—H1*w*1···O3$^{iii}$ | 0.9146 (10) | 2.0399 (10) | 2.9352 (14) | 165.84 (7) |
| O7—H7···O1 | 1.0191 (9) | 1.4329 (9) | 2.4340 (13) | 165.94 (7) |

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

**1-Aza-8-azoniabicyclo[5.4.0]undec-7-ene 4-aminobenzoate (WADXOR)**

*Crystal data*

$C_9H_{17}N_2^+ \cdot C_7H_3N_2O_7^-$
$M_r = 380.35$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yabc
$a = 6.1537$ (3) Å
$b = 19.1541$ (14) Å
$c = 14.5527$ (11) Å
$\beta = 98.343$ (6)°
$V = 1697.2$ (2) Å$^3$
$Z = 4$
$F(000) = 800$
$D_x = 1.489$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1891 reflections
$\theta = 3.5$–$26.6°$
$\mu = 0.12$ mm$^{-1}$
$T = 200$ K
Needle, yellow
0.30 × 0.13 × 0.10 mm

*Data collection*

Oxford Diffraction Gemini-S CCD-detector diffractometer
Graphite monochromator
Detector resolution: 16.067 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
$T_{min} = 0.920$, $T_{max} = 0.990$
7800 measured reflections
3339 independent reflections
1976 reflections with $I > 3\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.4°$
$h = -7 \rightarrow 7$
$k = -23 \rightarrow 23$
$l = -17 \rightarrow 17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.095$
$S = 1.33$
3339 reflections
268 parameters
2 restraints
98 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0004I^2)$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$
Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
Extinction coefficient: 2200 (700)

*Special details*

**Geometry**. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.
Number of fixed parameters : 10

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O2b | 0.8418 (3) | 0.56169 (11) | 0.78950 (14) | 0.0440 (8) | 0.727 (4) |
| H61b | 0.852107 | 0.560819 | 0.76707 | 0.0359* | 0.273 (4) |

| | | | | | |
|---|---|---|---|---|---|
| O21b | 0.7735 (8) | 0.6569 (3) | 0.4912 (4) | 0.048 (2) | 0.273 (4) |
| H6b | 0.805712 | 0.644967 | 0.507207 | 0.0328* | 0.727 (4) |
| O11b | 0.5083 (2) | 0.68884 (8) | 0.59278 (11) | 0.0444 (6) | |
| O12b | 0.5447 (2) | 0.64534 (8) | 0.73586 (12) | 0.0524 (7) | |
| O31b | 1.1108 (3) | 0.45706 (10) | 0.81717 (14) | 0.0829 (9) | |
| O32b | 1.4073 (3) | 0.47245 (10) | 0.75770 (13) | 0.0775 (8) | |
| O51b | 1.3288 (3) | 0.55869 (9) | 0.44602 (12) | 0.0603 (7) | |
| O52b | 1.0707 (3) | 0.63212 (10) | 0.39870 (12) | 0.0680 (8) | |
| N3b | 1.2111 (3) | 0.48284 (10) | 0.76049 (14) | 0.0472 (8) | |
| N5b | 1.1654 (3) | 0.59180 (10) | 0.45701 (14) | 0.0418 (7) | |
| C1b | 0.8002 (3) | 0.60956 (10) | 0.63903 (15) | 0.0264 (7) | |
| C2b | 0.9062 (3) | 0.56604 (11) | 0.70954 (15) | 0.0299 (7) | |
| C3b | 1.0952 (3) | 0.53063 (10) | 0.69140 (15) | 0.0309 (7) | |
| C4b | 1.1814 (3) | 0.53946 (10) | 0.61042 (15) | 0.0309 (7) | |
| C5b | 1.0736 (3) | 0.58234 (10) | 0.54290 (14) | 0.0271 (7) | |
| C6b | 0.8810 (3) | 0.61681 (10) | 0.55538 (15) | 0.0273 (7) | |
| C11b | 0.6030 (3) | 0.65080 (11) | 0.65583 (18) | 0.0351 (8) | |
| N1a | −0.1521 (2) | 0.82026 (8) | 0.63826 (12) | 0.0293 (6) | |
| N8a | 0.1721 (2) | 0.76019 (9) | 0.67283 (12) | 0.0373 (7) | |
| C2a | −0.3261 (3) | 0.85089 (11) | 0.57022 (15) | 0.0370 (8) | |
| C3a | −0.2602 (3) | 0.91805 (11) | 0.52679 (15) | 0.0401 (8) | |
| C4a | −0.1188 (3) | 0.90791 (11) | 0.45061 (16) | 0.0417 (8) | |
| C5a | 0.0933 (3) | 0.86805 (11) | 0.48032 (14) | 0.0401 (8) | |
| C6a | 0.0611 (3) | 0.79367 (11) | 0.51423 (14) | 0.0344 (8) | |
| C7a | 0.0226 (3) | 0.79087 (10) | 0.61288 (14) | 0.0268 (7) | |
| C9a | 0.1394 (8) | 0.7480 (3) | 0.7694 (4) | 0.0377 (17) | 0.687 (4) |
| C10a | 0.0234 (5) | 0.8112 (2) | 0.8006 (2) | 0.0388 (12) | 0.687 (4) |
| C11a | −0.1865 (3) | 0.82334 (13) | 0.73606 (16) | 0.0375 (8) | |
| C13a | 0.192 (2) | 0.7733 (6) | 0.7752 (10) | 0.0377 (17) | 0.313 (4) |
| C12a | −0.0453 (12) | 0.7700 (5) | 0.7962 (6) | 0.0388 (12) | 0.313 (4) |
| H4b | 1.313123 | 0.516393 | 0.601144 | 0.0371* | |
| H8a | 0.304017 | 0.743029 | 0.652706 | 0.073 (8)* | |
| H10a | 0.119515 | 0.852593 | 0.800333 | 0.0466* | 0.686 |
| H21a | −0.458393 | 0.859249 | 0.600103 | 0.0444* | |
| H22a | −0.373911 | 0.816372 | 0.520773 | 0.0444* | |
| H31a | −0.393523 | 0.944805 | 0.502041 | 0.0481* | |
| H32a | −0.182575 | 0.948658 | 0.575716 | 0.0481* | |
| H41a | −0.084564 | 0.95395 | 0.425285 | 0.0501* | |
| H42a | −0.20539 | 0.883982 | 0.397103 | 0.0501* | |
| H51a | 0.180506 | 0.866759 | 0.428286 | 0.0482* | |
| H52a | 0.186838 | 0.894538 | 0.52943 | 0.0482* | |
| H61a | 0.191356 | 0.765112 | 0.506561 | 0.0413* | |
| H62a | −0.063947 | 0.77162 | 0.474177 | 0.0413* | |
| H91a | 0.046176 | 0.706149 | 0.772504 | 0.0452* | 0.687 (4) |
| H92a | 0.283833 | 0.74265 | 0.808712 | 0.0452* | 0.687 (4) |
| H11a | −0.009873 | 0.803439 | 0.864351 | 0.0466* | 0.686 |
| H12a | −0.318 (2) | 0.7965 (9) | 0.7425 (13) | 0.045* | |
| H13a | −0.230 (3) | 0.8711 (6) | 0.7485 (13) | 0.045* | |

| | | | | | |
|---|---|---|---|---|---|
| H14a | −0.105082 | 0.7226 | 0.782436 | 0.0466* | 0.313 (4) |
| H15a | −0.047311 | 0.780768 | 0.862598 | 0.0466* | 0.313 (4) |
| H16a | 0.27893 | 0.735519 | 0.809131 | 0.0452* | 0.313 (4) |
| H17a | 0.251697 | 0.820686 | 0.789237 | 0.0452* | 0.313 (4) |
| H21b | 0.621837 | 0.678052 | 0.538628 | 0.082 (11)* | 0.273 (4) |
| H2b | 0.702896 | 0.598291 | 0.774475 | 0.082 (11)* | 0.727 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O2b | 0.0481 (12) | 0.0570 (16) | 0.0296 (15) | 0.0087 (11) | 0.0152 (10) | 0.0091 (11) |
| O21b | 0.045 (3) | 0.053 (4) | 0.046 (4) | 0.006 (3) | 0.006 (3) | 0.023 (3) |
| O11b | 0.0369 (8) | 0.0391 (10) | 0.0583 (12) | 0.0136 (7) | 0.0102 (8) | 0.0073 (9) |
| O12b | 0.0488 (9) | 0.0643 (12) | 0.0488 (12) | 0.0091 (8) | 0.0232 (8) | −0.0030 (9) |
| O31b | 0.0796 (13) | 0.0853 (16) | 0.0753 (16) | −0.0256 (11) | −0.0175 (11) | 0.0533 (13) |
| O32b | 0.0728 (12) | 0.0903 (16) | 0.0642 (14) | 0.0483 (11) | −0.0074 (10) | 0.0050 (11) |
| O51b | 0.0542 (10) | 0.0650 (12) | 0.0694 (13) | 0.0077 (9) | 0.0350 (9) | −0.0110 (10) |
| O52b | 0.0926 (13) | 0.0722 (14) | 0.0455 (12) | 0.0171 (11) | 0.0314 (10) | 0.0241 (10) |
| N3b | 0.0575 (13) | 0.0347 (13) | 0.0432 (15) | 0.0005 (11) | −0.0137 (11) | −0.0004 (10) |
| N5b | 0.0476 (11) | 0.0400 (13) | 0.0414 (13) | −0.0046 (10) | 0.0193 (10) | −0.0067 (10) |
| C1b | 0.0265 (10) | 0.0220 (11) | 0.0303 (13) | −0.0029 (9) | 0.0024 (9) | −0.0023 (10) |
| C2b | 0.0337 (11) | 0.0267 (12) | 0.0294 (14) | −0.0061 (10) | 0.0050 (10) | −0.0023 (10) |
| C3b | 0.0361 (11) | 0.0233 (12) | 0.0304 (14) | 0.0002 (9) | −0.0053 (10) | 0.0025 (10) |
| C4b | 0.0257 (10) | 0.0240 (12) | 0.0413 (15) | −0.0013 (9) | −0.0005 (10) | −0.0053 (11) |
| C5b | 0.0295 (10) | 0.0254 (12) | 0.0274 (13) | −0.0052 (9) | 0.0073 (9) | −0.0017 (10) |
| C6b | 0.0298 (10) | 0.0218 (12) | 0.0290 (13) | −0.0028 (9) | −0.0003 (9) | 0.0007 (10) |
| C11b | 0.0297 (11) | 0.0296 (13) | 0.0464 (16) | −0.0042 (10) | 0.0070 (11) | −0.0061 (12) |
| N1a | 0.0249 (8) | 0.0327 (11) | 0.0305 (11) | 0.0026 (8) | 0.0041 (8) | 0.0000 (9) |
| N8a | 0.0332 (9) | 0.0493 (12) | 0.0291 (12) | 0.0137 (9) | 0.0037 (8) | 0.0056 (9) |
| C2a | 0.0243 (10) | 0.0400 (14) | 0.0451 (15) | 0.0063 (10) | −0.0002 (10) | −0.0020 (12) |
| C3a | 0.0374 (12) | 0.0331 (14) | 0.0465 (16) | 0.0071 (10) | −0.0044 (11) | 0.0000 (11) |
| C4a | 0.0450 (12) | 0.0397 (15) | 0.0370 (15) | −0.0028 (11) | −0.0060 (11) | 0.0087 (11) |
| C5a | 0.0368 (11) | 0.0526 (16) | 0.0319 (14) | −0.0008 (11) | 0.0079 (10) | 0.0091 (12) |
| C6a | 0.0355 (11) | 0.0411 (15) | 0.0262 (13) | 0.0079 (10) | 0.0032 (9) | −0.0057 (11) |
| C7a | 0.0283 (10) | 0.0224 (12) | 0.0291 (13) | −0.0012 (9) | 0.0025 (9) | −0.0030 (10) |
| C9a | 0.040 (3) | 0.041 (4) | 0.0304 (18) | −0.001 (2) | −0.001 (2) | 0.004 (3) |
| C10a | 0.049 (2) | 0.042 (2) | 0.0270 (17) | −0.0043 (18) | 0.0083 (15) | −0.0029 (19) |
| C11a | 0.0367 (12) | 0.0426 (15) | 0.0364 (15) | 0.0003 (11) | 0.0161 (11) | −0.0056 (12) |
| C13a | 0.040 (3) | 0.041 (4) | 0.0304 (18) | −0.001 (2) | −0.001 (2) | 0.004 (3) |
| C12a | 0.049 (2) | 0.042 (2) | 0.0270 (17) | −0.0043 (18) | 0.0083 (15) | −0.0029 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O2b—C2b | 1.285 (3) | C2a—H22a | 0.99 |
| O2b—H2b | 1.103 (2) | C3a—C4a | 1.518 (3) |
| H61b—C2b | 0.95 | C3a—H31a | 0.99 |
| O21b—C6b | 1.311 (6) | C3a—H32a | 0.99 |
| O21b—H21b | 1.303 (6) | C4a—C5a | 1.520 (3) |

| | | | |
|---|---|---|---|
| H6b—C6b | 0.95 | C4a—H41a | 0.99 |
| O11b—C11b | 1.248 (3) | C4a—H42a | 0.99 |
| O11b—H21b | 1.1454 (16) | C5a—C6a | 1.530 (3) |
| O12b—C11b | 1.272 (3) | C5a—H51a | 0.99 |
| O12b—H2b | 1.3846 (15) | C5a—H52a | 0.99 |
| O31b—N3b | 1.205 (3) | C6a—C7a | 1.490 (3) |
| O32b—N3b | 1.230 (3) | C6a—H61a | 0.99 |
| O51b—N5b | 1.219 (3) | C6a—H62a | 0.99 |
| O52b—N5b | 1.230 (3) | C9a—C10a | 1.508 (6) |
| N3b—C3b | 1.466 (3) | C9a—C13a | 0.581 (13) |
| N5b—C5b | 1.456 (3) | C9a—C12a | 1.323 (10) |
| C1b—C2b | 1.407 (3) | C9a—H91a | 0.99 |
| C1b—C6b | 1.387 (3) | C9a—H92a | 0.99 |
| C1b—C11b | 1.497 (3) | C9a—H16a | 0.9922 |
| C2b—C3b | 1.404 (3) | C10a—C11a | 1.501 (4) |
| C3b—C4b | 1.371 (3) | C10a—C13a | 1.358 (14) |
| C4b—C5b | 1.375 (3) | C10a—H10a | 0.99 |
| C4b—H4b | 0.95 | C10a—H11a | 0.99 |
| C5b—C6b | 1.392 (3) | C10a—H15a | 1.207 |
| N1a—C2a | 1.471 (2) | C11a—C12a | 1.530 (8) |
| N1a—C7a | 1.313 (2) | C11a—H12a | 0.977 (15) |
| N1a—C11a | 1.470 (3) | C11a—H13a | 0.978 (13) |
| N8a—C7a | 1.311 (2) | C13a—C12a | 1.534 (16) |
| N8a—C9a | 1.467 (6) | C13a—H92a | 0.9083 |
| N8a—C13a | 1.498 (15) | C13a—H16a | 0.99 |
| N8a—H8a | 0.9604 (17) | C13a—H17a | 0.99 |
| C2a—C3a | 1.514 (3) | C12a—H14a | 0.99 |
| C2a—H21a | 0.99 | C12a—H15a | 0.99 |

| | | | |
|---|---|---|---|
| H6b—O21b—H21b | 102.88 | N8a—C9a—C13a | 81.6 (16) |
| C11b—O12b—H2b | 98.61 (14) | N8a—C9a—C12a | 118.3 (5) |
| O31b—N3b—O32b | 124.0 (2) | N8a—C9a—H91a | 109.47 |
| O31b—N3b—C3b | 118.6 (2) | N8a—C9a—H92a | 109.47 |
| O32b—N3b—C3b | 117.4 (2) | N8a—C9a—H14a | 114.78 |
| O51b—N5b—O52b | 123.6 (2) | N8a—C9a—H16a | 111.65 |
| O51b—N5b—C5b | 118.57 (18) | C10a—C9a—H91a | 109.47 |
| O52b—N5b—C5b | 117.83 (18) | C10a—C9a—H92a | 109.47 |
| C2b—C1b—C6b | 120.82 (17) | C13a—C9a—C12a | 99.9 (15) |
| C2b—C1b—C11b | 119.6 (2) | C13a—C9a—H91a | 168.74 |
| C6b—C1b—C11b | 119.51 (18) | C13a—C9a—H14a | 137.56 |
| O2b—C2b—C1b | 121.84 (19) | C12a—C9a—H92a | 126.91 |
| O2b—C2b—C3b | 120.77 (19) | C12a—C9a—H16a | 127.82 |
| H61b—C2b—C1b | 121.37 | H91a—C9a—H92a | 111.59 |
| H61b—C2b—C3b | 121.37 | H14a—C9a—H16a | 126.91 |
| C1b—C2b—C3b | 117.3 (2) | H14a—C9a—H17a | 129.78 |
| N3b—C3b—C2b | 120.4 (2) | H16a—C9a—H17a | 77.55 |
| N3b—C3b—C4b | 117.12 (18) | C9a—C10a—C11a | 109.8 (3) |
| C2b—C3b—C4b | 122.44 (18) | C11a—C10a—C13a | 122.2 (6) |

| | | | |
|---|---|---|---|
| C3b—C4b—C5b | 118.75 (18) | C11a—C10a—H10a | 109.47 |
| C3b—C4b—H4b | 120.62 | C11a—C10a—H11a | 109.47 |
| C5b—C4b—H4b | 120.62 | C11a—C10a—H17a | 132.02 |
| N5b—C5b—C4b | 118.74 (17) | C13a—C10a—H11a | 116.43 |
| N5b—C5b—C6b | 119.80 (17) | C13a—C10a—H15a | 108.54 |
| C4b—C5b—C6b | 121.5 (2) | C12a—C10a—H17a | 124.07 |
| O21b—C6b—C1b | 118.4 (3) | H10a—C10a—H11a | 109.16 |
| O21b—C6b—C5b | 122.4 (3) | H10a—C10a—H15a | 132.06 |
| H6b—C6b—C1b | 120.41 | H11a—C10a—H17a | 117.48 |
| H6b—C6b—C5b | 120.41 | H15a—C10a—H17a | 127.34 |
| C1b—C6b—C5b | 119.17 (18) | N1a—C11a—C10a | 111.6 (2) |
| O11b—C11b—O12b | 123.9 (2) | N1a—C11a—C12a | 112.2 (4) |
| O11b—C11b—C1b | 119.4 (2) | N1a—C11a—H12a | 108.1 (11) |
| O12b—C11b—C1b | 116.70 (19) | N1a—C11a—H13a | 107.3 (12) |
| C2a—N1a—C7a | 121.83 (17) | C10a—C11a—H12a | 120.7 (10) |
| C2a—N1a—C11a | 116.29 (16) | C10a—C11a—H13a | 105.2 (10) |
| C7a—N1a—C11a | 121.87 (16) | C12a—C11a—H13a | 131.8 (11) |
| C7a—N8a—C9a | 121.9 (2) | H12a—C11a—H13a | 102.9 (15) |
| C7a—N8a—C13a | 122.3 (5) | N8a—C13a—C9a | 75.8 (15) |
| C7a—N8a—H8a | 119.53 (19) | N8a—C13a—C10a | 114.0 (9) |
| C9a—N8a—C13a | 22.6 (5) | N8a—C13a—C12a | 104.5 (8) |
| C9a—N8a—H8a | 118.5 (2) | N8a—C13a—H10a | 113.12 |
| C13a—N8a—H8a | 114.3 (5) | N8a—C13a—H92a | 112.32 |
| N1a—C2a—C3a | 114.01 (15) | N8a—C13a—H16a | 109.47 |
| N1a—C2a—H21a | 109.47 | N8a—C13a—H17a | 109.47 |
| N1a—C2a—H22a | 109.47 | C9a—C13a—H10a | 130.08 |
| C3a—C2a—H21a | 109.47 | C9a—C13a—H17a | 167.66 |
| C3a—C2a—H22a | 109.47 | C10a—C13a—H92a | 129.79 |
| H21a—C2a—H22a | 104.52 | C10a—C13a—H16a | 129.35 |
| C2a—C3a—C4a | 114.39 (18) | C12a—C13a—H92a | 113.84 |
| C2a—C3a—H31a | 109.47 | C12a—C13a—H16a | 109.47 |
| C2a—C3a—H32a | 109.47 | C12a—C13a—H17a | 109.47 |
| C4a—C3a—H31a | 109.47 | H10a—C13a—H91a | 126.53 |
| C4a—C3a—H32a | 109.47 | H10a—C13a—H92a | 130.35 |
| H31a—C3a—H32a | 104.05 | H10a—C13a—H16a | 135.32 |
| C3a—C4a—C5a | 114.58 (18) | H92a—C13a—H17a | 107.21 |
| C3a—C4a—H41a | 109.47 | H16a—C13a—H17a | 114.05 |
| C3a—C4a—H42a | 109.47 | C9a—C12a—C11a | 119.1 (6) |
| C5a—C4a—H41a | 109.47 | C9a—C12a—H11a | 111.16 |
| C5a—C4a—H42a | 109.47 | C9a—C12a—H15a | 119.13 |
| H41a—C4a—H42a | 103.83 | C10a—C12a—H91a | 125.59 |
| C4a—C5a—C6a | 114.44 (16) | C10a—C12a—H14a | 169.66 |
| C4a—C5a—H51a | 109.47 | C11a—C12a—C13a | 109.6 (7) |
| C4a—C5a—H52a | 109.47 | C11a—C12a—H91a | 130.75 |
| C6a—C5a—H51a | 109.47 | C11a—C12a—H14a | 109.47 |
| C6a—C5a—H52a | 109.47 | C11a—C12a—H15a | 109.47 |
| H51a—C5a—H52a | 104 | C13a—C12a—H14a | 109.47 |
| C5a—C6a—C7a | 112.99 (17) | C13a—C12a—H15a | 109.47 |

| | | | |
|---|---|---|---|
| C5a—C6a—H61a | 109.47 | H91a—C12a—H11a | 130.35 |
| C5a—C6a—H62a | 109.47 | H91a—C12a—H15a | 118.71 |
| C7a—C6a—H61a | 109.47 | H11a—C12a—H14a | 133.54 |
| C7a—C6a—H62a | 109.47 | H14a—C12a—H15a | 109.31 |
| H61a—C6a—H62a | 105.71 | O21b—H21b—O11b | 167.3 (3) |
| N1a—C7a—N8a | 121.93 (19) | H6b—H21b—O11b | 152.98 |
| N1a—C7a—C6a | 120.45 (16) | O2b—H2b—O12b | 166.81 (13) |
| N8a—C7a—C6a | 117.59 (17) | H61b—H2b—O12b | 150.18 |
| N8a—C9a—C10a | 107.3 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N8*a*—H8*a*···O11*b* | 0.9604 (17) | 1.9329 (15) | 2.864 (2) | 162.83 (11) |
| C10*a*—H10*a*···O32*b*$^{i}$ | 0.99 | 2.44 | 3.248 (4) | 138 |
| C2*a*—H21*a*···O31*b*$^{ii}$ | 0.99 | 2.48 | 3.275 (3) | 137 |
| C6*a*—H62*a*···O21*b*$^{iii}$ | 0.99 | 2.44 | 3.152 (6) | 128 |
| C10*a*—H11*a*···O21*b*$^{iv}$ | 0.99 | 2.47 | 3.033 (7) | 116 |
| O21*b*—H21*b*···O11*b* | 1.303 (6) | 1.1454 (16) | 2.433 (6) | 167.3 (3) |
| O11*b*—H21*b*···O21*b* | 1.1454 (16) | 1.303 (6) | 2.433 (6) | 167.3 (3) |
| O12*b*—H2*b*···O2*b* | 1.3846 (15) | 1.103 (2) | 2.471 (2) | 166.81 (13) |

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+3/2; (ii) −*x*+1/2, *y*+1/2, −*z*+3/2; (iii) *x*−1, *y*, *z*; (iv) *x*−1/2, −*y*+3/2, *z*+1/2.

## 4-(Diphenylmethyl)-1-(3-phenylprop-2-en-1-yl)piperazin-1-ium 2-carboxy-4,6-dinitrophenolate (YAXPOE)

*Crystal data*

$C_{26}H_{29}N_2^+·C_7H_3N_2O_7^-$
$M_r$ = 596.63
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
*a* = 14.5648 (3) Å
*b* = 12.9374 (3) Å
*c* = 16.1619 (3) Å
*β* = 103.900 (1)°
*V* = 2956.22 (11) Å$^3$
*Z* = 4
*F*(000) = 1256
$D_x$ = 1.341 Mg m$^{-3}$
Melting point: 383 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9909 reflections
*θ* = 2.3–28.3°
*μ* = 0.10 mm$^{-1}$
*T* = 200 K
Block, yellow
0.51 × 0.26 × 0.17 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*φ* and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min}$ = 0.932, $T_{max}$ = 1.000
29552 measured reflections
7344 independent reflections
5724 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.015
$\theta_{max}$ = 28.3°, $\theta_{min}$ = 2.0°
*h* = −19→19
*k* = −17→17
*l* = −21→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F > 3\sigma(F)] = 0.054$
$wR(F) = 0.190$
$S = 1.80$
7344 reflections
399 parameters
0 restraints
120 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Weighting scheme based on measured s.u.'s $w = 1/(\sigma^2(I) + 0.0063999998I^2)$
$(\Delta/\sigma)_{max} = 0.044$
$\Delta\rho_{max} = 0.63$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

*Special details*

**Refinement**. Number of fixed parameters 6

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.25627 (8) | 0.71181 (9) | 0.31343 (7) | 0.0246 (3) |
| H71 | 0.30128 | 0.669474 | 0.349979 | 0.035 (4)* |
| N2 | 0.10023 (8) | 0.56683 (9) | 0.26575 (7) | 0.0233 (3) |
| C1 | 0.29865 (11) | 0.95151 (12) | 0.41661 (10) | 0.0334 (5) |
| H1a | 0.245898 | 0.980932 | 0.377422 | 0.0401* |
| C2 | 0.33463 (11) | 0.86695 (12) | 0.39170 (10) | 0.0322 (5) |
| H2 | 0.386765 | 0.835478 | 0.430202 | 0.0386* |
| C3 | 0.29839 (12) | 0.81754 (12) | 0.30656 (10) | 0.0333 (5) |
| H3a | 0.350521 | 0.811391 | 0.27725 | 0.04* |
| H3b | 0.250048 | 0.862741 | 0.270514 | 0.04* |
| C4 | 0.17112 (10) | 0.71678 (11) | 0.34935 (9) | 0.0281 (4) |
| H4a | 0.123241 | 0.762704 | 0.313566 | 0.0337* |
| H4b | 0.189039 | 0.745897 | 0.40767 | 0.0337* |
| C5 | 0.12943 (10) | 0.61031 (11) | 0.35226 (9) | 0.0271 (4) |
| H5a | 0.07387 | 0.614805 | 0.377337 | 0.0326* |
| H5b | 0.177161 | 0.564522 | 0.388386 | 0.0326* |
| C6 | 0.18571 (10) | 0.55563 (11) | 0.23345 (9) | 0.0262 (4) |
| H6a | 0.231171 | 0.509606 | 0.271816 | 0.0315* |
| H6b | 0.168931 | 0.5233 | 0.176272 | 0.0315* |
| C7 | 0.23149 (10) | 0.65944 (12) | 0.22789 (9) | 0.0282 (4) |
| H7a | 0.289537 | 0.649813 | 0.207068 | 0.0338* |
| H7b | 0.187639 | 0.703827 | 0.186493 | 0.0338* |
| C8 | 0.05326 (9) | 0.46528 (10) | 0.26756 (8) | 0.0230 (4) |
| H8 | 0.099282 | 0.417266 | 0.304542 | 0.0276* |
| C11 | 0.33145 (11) | 1.00535 (12) | 0.49876 (10) | 0.0326 (5) |
| C12 | 0.41578 (13) | 0.97941 (14) | 0.55685 (10) | 0.0391 (5) |
| H12 | 0.454927 | 0.926517 | 0.543205 | 0.047* |
| C13 | 0.44278 (15) | 1.03053 (17) | 0.63457 (11) | 0.0502 (6) |
| H13 | 0.499799 | 1.011658 | 0.674094 | 0.0603* |
| C14 | 0.38752 (17) | 1.10810 (17) | 0.65451 (12) | 0.0554 (7) |

| | | | | |
|---|---|---|---|---|
| H14 | 0.40688 | 1.143158 | 0.707495 | 0.0665* |
| C15 | 0.30389 (15) | 1.13552 (16) | 0.59798 (13) | 0.0516 (7) |
| H15 | 0.265849 | 1.18931 | 0.61197 | 0.0619* |
| C16 | 0.27570 (13) | 1.08400 (13) | 0.52053 (11) | 0.0397 (5) |
| H16 | 0.217882 | 1.102436 | 0.481949 | 0.0476* |
| C21 | −0.03166 (9) | 0.47504 (10) | 0.30647 (8) | 0.0240 (4) |
| C22 | −0.04272 (11) | 0.40385 (12) | 0.36763 (10) | 0.0315 (5) |
| H22 | 0.003276 | 0.351198 | 0.385315 | 0.0378* |
| C23 | −0.12077 (12) | 0.40899 (13) | 0.40333 (11) | 0.0388 (5) |
| H23 | −0.127745 | 0.359826 | 0.445056 | 0.0466* |
| C24 | −0.18805 (12) | 0.48544 (13) | 0.37818 (11) | 0.0385 (5) |
| H24 | −0.241677 | 0.488365 | 0.401911 | 0.0461* |
| C25 | −0.17687 (11) | 0.55719 (12) | 0.31859 (11) | 0.0351 (5) |
| H25 | −0.222711 | 0.610174 | 0.301738 | 0.0421* |
| C26 | −0.09910 (10) | 0.55304 (11) | 0.28271 (9) | 0.0286 (4) |
| H26 | −0.091899 | 0.603396 | 0.241958 | 0.0344* |
| C31 | 0.02520 (10) | 0.41880 (11) | 0.17810 (9) | 0.0248 (4) |
| C32 | 0.06690 (11) | 0.32720 (12) | 0.16027 (10) | 0.0331 (5) |
| H32 | 0.112496 | 0.2938 | 0.204043 | 0.0397* |
| C33 | 0.04254 (13) | 0.28416 (13) | 0.07918 (11) | 0.0392 (5) |
| H33 | 0.071842 | 0.221893 | 0.067798 | 0.047* |
| C34 | −0.02351 (12) | 0.33108 (13) | 0.01557 (10) | 0.0379 (5) |
| H34 | −0.039638 | 0.301886 | −0.039979 | 0.0455* |
| C35 | −0.06672 (12) | 0.42142 (14) | 0.03267 (10) | 0.0385 (5) |
| H35 | −0.113709 | 0.453238 | −0.01092 | 0.0461* |
| C36 | −0.04162 (11) | 0.46550 (12) | 0.11316 (10) | 0.0321 (5) |
| H36 | −0.070483 | 0.528311 | 0.123929 | 0.0386* |
| O1 | 0.63327 (8) | 0.37352 (10) | 0.53505 (8) | 0.0392 (4) |
| O2 | 0.58197 (10) | 0.44006 (14) | 0.67133 (9) | 0.0614 (6) |
| O3 | 0.43608 (10) | 0.48129 (12) | 0.65684 (9) | 0.0549 (5) |
| O4 | 0.19464 (9) | 0.32143 (13) | 0.44743 (10) | 0.0569 (5) |
| O5 | 0.22764 (11) | 0.24248 (13) | 0.34091 (10) | 0.0631 (6) |
| O6 | 0.54745 (10) | 0.23529 (12) | 0.30214 (8) | 0.0518 (5) |
| O7 | 0.66627 (9) | 0.28940 (12) | 0.40564 (9) | 0.0507 (5) |
| N3 | 0.49861 (10) | 0.43835 (11) | 0.63197 (9) | 0.0379 (4) |
| N4 | 0.25133 (10) | 0.29121 (12) | 0.40756 (10) | 0.0428 (5) |
| C9 | 0.57563 (13) | 0.27360 (13) | 0.37128 (12) | 0.0398 (6) |
| C41 | 0.54458 (10) | 0.35627 (11) | 0.50735 (10) | 0.0298 (4) |
| C42 | 0.50987 (11) | 0.30564 (12) | 0.42622 (10) | 0.0314 (5) |
| C43 | 0.41587 (11) | 0.28489 (12) | 0.39522 (10) | 0.0329 (5) |
| H43 | 0.395087 | 0.249901 | 0.342348 | 0.0394* |
| C44 | 0.35106 (10) | 0.31453 (12) | 0.44039 (10) | 0.0313 (4) |
| C45 | 0.37850 (10) | 0.36477 (12) | 0.51759 (10) | 0.0305 (4) |
| H45 | 0.332901 | 0.38543 | 0.547596 | 0.0366* |
| C46 | 0.47336 (11) | 0.38456 (11) | 0.55054 (9) | 0.0290 (4) |
| H7 | 0.676841 | 0.321683 | 0.458063 | 0.030 (4)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0221 (6) | 0.0282 (6) | 0.0236 (5) | −0.0039 (4) | 0.0055 (4) | −0.0028 (4) |
| N2 | 0.0209 (5) | 0.0268 (6) | 0.0227 (5) | −0.0035 (4) | 0.0063 (4) | −0.0032 (4) |
| C1 | 0.0300 (8) | 0.0358 (8) | 0.0314 (7) | −0.0037 (6) | 0.0017 (6) | −0.0003 (6) |
| C2 | 0.0301 (7) | 0.0308 (7) | 0.0334 (7) | −0.0076 (6) | 0.0033 (6) | 0.0004 (6) |
| C3 | 0.0391 (8) | 0.0304 (7) | 0.0318 (7) | −0.0113 (6) | 0.0111 (6) | −0.0022 (6) |
| C4 | 0.0216 (6) | 0.0331 (7) | 0.0302 (7) | −0.0026 (5) | 0.0075 (5) | −0.0076 (5) |
| C5 | 0.0248 (7) | 0.0337 (7) | 0.0241 (6) | −0.0051 (5) | 0.0082 (5) | −0.0053 (5) |
| C6 | 0.0232 (6) | 0.0304 (7) | 0.0265 (6) | −0.0032 (5) | 0.0088 (5) | −0.0047 (5) |
| C7 | 0.0285 (7) | 0.0344 (7) | 0.0223 (6) | −0.0060 (6) | 0.0073 (5) | −0.0045 (5) |
| C8 | 0.0197 (6) | 0.0247 (6) | 0.0245 (6) | 0.0003 (5) | 0.0054 (5) | 0.0010 (5) |
| C11 | 0.0357 (8) | 0.0306 (7) | 0.0307 (7) | −0.0080 (6) | 0.0064 (6) | 0.0006 (6) |
| C12 | 0.0398 (9) | 0.0415 (9) | 0.0337 (8) | −0.0067 (7) | 0.0042 (7) | 0.0007 (7) |
| C13 | 0.0523 (11) | 0.0604 (12) | 0.0319 (8) | −0.0142 (9) | −0.0018 (8) | 0.0005 (8) |
| C14 | 0.0691 (14) | 0.0629 (13) | 0.0354 (9) | −0.0230 (10) | 0.0149 (9) | −0.0159 (8) |
| C15 | 0.0584 (12) | 0.0455 (10) | 0.0562 (11) | −0.0106 (9) | 0.0242 (10) | −0.0151 (8) |
| C16 | 0.0392 (9) | 0.0350 (8) | 0.0444 (9) | −0.0049 (7) | 0.0090 (7) | −0.0039 (7) |
| C21 | 0.0213 (6) | 0.0250 (6) | 0.0255 (6) | −0.0020 (5) | 0.0050 (5) | −0.0024 (5) |
| C22 | 0.0311 (7) | 0.0329 (8) | 0.0325 (7) | 0.0030 (6) | 0.0114 (6) | 0.0046 (6) |
| C23 | 0.0426 (9) | 0.0390 (9) | 0.0413 (9) | 0.0005 (7) | 0.0228 (7) | 0.0058 (7) |
| C24 | 0.0344 (8) | 0.0391 (8) | 0.0493 (9) | −0.0028 (6) | 0.0245 (7) | −0.0078 (7) |
| C25 | 0.0278 (7) | 0.0336 (8) | 0.0447 (9) | 0.0044 (6) | 0.0104 (6) | −0.0058 (6) |
| C26 | 0.0269 (7) | 0.0271 (7) | 0.0323 (7) | 0.0015 (5) | 0.0079 (6) | 0.0002 (5) |
| C31 | 0.0226 (6) | 0.0253 (6) | 0.0282 (6) | −0.0049 (5) | 0.0093 (5) | −0.0016 (5) |
| C32 | 0.0327 (8) | 0.0296 (7) | 0.0367 (8) | 0.0017 (6) | 0.0076 (6) | −0.0021 (6) |
| C33 | 0.0437 (9) | 0.0299 (8) | 0.0463 (9) | −0.0014 (7) | 0.0154 (8) | −0.0097 (7) |
| C34 | 0.0452 (9) | 0.0381 (9) | 0.0313 (8) | −0.0104 (7) | 0.0109 (7) | −0.0090 (6) |
| C35 | 0.0387 (9) | 0.0451 (9) | 0.0283 (7) | −0.0015 (7) | 0.0017 (7) | −0.0024 (7) |
| C36 | 0.0316 (8) | 0.0337 (8) | 0.0310 (7) | 0.0020 (6) | 0.0073 (6) | −0.0020 (6) |
| O1 | 0.0242 (6) | 0.0466 (7) | 0.0449 (6) | −0.0022 (5) | 0.0046 (5) | 0.0065 (5) |
| O2 | 0.0364 (7) | 0.0954 (12) | 0.0457 (8) | 0.0021 (7) | −0.0029 (6) | −0.0243 (7) |
| O3 | 0.0459 (8) | 0.0698 (9) | 0.0453 (7) | 0.0153 (7) | 0.0038 (6) | −0.0171 (6) |
| O4 | 0.0239 (6) | 0.0732 (10) | 0.0713 (9) | 0.0049 (6) | 0.0067 (6) | −0.0008 (8) |
| O5 | 0.0465 (8) | 0.0774 (11) | 0.0531 (8) | −0.0103 (7) | −0.0120 (7) | −0.0150 (7) |
| O6 | 0.0595 (9) | 0.0612 (9) | 0.0400 (7) | 0.0066 (7) | 0.0226 (6) | −0.0026 (6) |
| O7 | 0.0391 (7) | 0.0619 (9) | 0.0567 (8) | 0.0007 (6) | 0.0223 (6) | −0.0035 (6) |
| N3 | 0.0355 (7) | 0.0405 (8) | 0.0347 (7) | 0.0038 (6) | 0.0023 (6) | −0.0023 (6) |
| N4 | 0.0303 (7) | 0.0466 (8) | 0.0442 (8) | −0.0012 (6) | −0.0056 (6) | 0.0040 (6) |
| C9 | 0.0393 (9) | 0.0352 (8) | 0.0491 (10) | 0.0046 (7) | 0.0190 (8) | 0.0095 (7) |
| C41 | 0.0232 (7) | 0.0277 (7) | 0.0368 (7) | 0.0028 (5) | 0.0038 (6) | 0.0099 (6) |
| C42 | 0.0330 (8) | 0.0293 (7) | 0.0345 (7) | 0.0049 (6) | 0.0133 (6) | 0.0074 (6) |
| C43 | 0.0370 (8) | 0.0322 (8) | 0.0284 (7) | 0.0011 (6) | 0.0059 (6) | 0.0030 (6) |
| C44 | 0.0238 (7) | 0.0334 (8) | 0.0333 (7) | 0.0012 (5) | 0.0000 (6) | 0.0037 (6) |
| C45 | 0.0255 (7) | 0.0325 (7) | 0.0333 (7) | 0.0060 (6) | 0.0067 (6) | 0.0030 (6) |
| C46 | 0.0282 (7) | 0.0282 (7) | 0.0282 (7) | 0.0018 (5) | 0.0017 (6) | 0.0016 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—H71 | 0.9448 (11) | C16—H16 | 0.95 |
| N1—C3 | 1.5141 (19) | C21—C22 | 1.388 (2) |
| N1—C4 | 1.492 (2) | C21—C26 | 1.3962 (19) |
| N1—C7 | 1.5036 (18) | C22—H22 | 0.95 |
| N2—C5 | 1.4718 (17) | C22—C23 | 1.395 (3) |
| N2—C6 | 1.4682 (19) | C23—H23 | 0.95 |
| N2—C8 | 1.4848 (17) | C23—C24 | 1.383 (2) |
| C1—H1a | 0.95 | C24—H24 | 0.95 |
| C1—C2 | 1.317 (2) | C24—C25 | 1.375 (3) |
| C1—C11 | 1.474 (2) | C25—H25 | 0.95 |
| C2—H2 | 0.95 | C25—C26 | 1.392 (2) |
| C2—C3 | 1.494 (2) | C26—H26 | 0.95 |
| C3—H3a | 0.99 | C31—C32 | 1.393 (2) |
| C3—H3b | 0.99 | C31—C36 | 1.3867 (19) |
| H3a—H3b | 1.5869 | C32—H32 | 0.95 |
| C4—H4a | 0.99 | C32—C33 | 1.389 (2) |
| C4—H4b | 0.99 | C33—H33 | 0.95 |
| C4—C5 | 1.511 (2) | C33—C34 | 1.370 (2) |
| H4a—H4b | 1.6058 | C34—H34 | 0.95 |
| C5—H5a | 0.99 | C34—C35 | 1.386 (3) |
| C5—H5b | 0.99 | C35—H35 | 0.95 |
| H5a—H5b | 1.6091 | C35—C36 | 1.387 (2) |
| C6—H6a | 0.99 | C36—H36 | 0.95 |
| C6—H6b | 0.99 | O1—C41 | 1.2810 (17) |
| C6—C7 | 1.512 (2) | O2—N3 | 1.2281 (18) |
| H6a—H6b | 1.6016 | O3—N3 | 1.215 (2) |
| C7—H7a | 0.99 | O4—N4 | 1.227 (2) |
| C7—H7b | 0.99 | O5—N4 | 1.224 (2) |
| H7a—H7b | 1.6014 | O6—C9 | 1.201 (2) |
| C8—H8 | 1 | O7—C9 | 1.319 (2) |
| C8—C21 | 1.522 (2) | O7—H7 | 0.9238 (15) |
| C8—C31 | 1.5282 (19) | N3—C46 | 1.456 (2) |
| C11—C12 | 1.396 (2) | N4—C44 | 1.453 (2) |
| C11—C16 | 1.399 (3) | C9—C42 | 1.512 (3) |
| C12—H12 | 0.95 | C41—C42 | 1.444 (2) |
| C12—C13 | 1.390 (2) | C41—C46 | 1.430 (2) |
| C13—H13 | 0.95 | C42—C43 | 1.367 (2) |
| C13—C14 | 1.372 (3) | C43—H43 | 0.95 |
| C14—H14 | 0.95 | C43—C44 | 1.379 (2) |
| C14—C15 | 1.382 (3) | C44—C45 | 1.378 (2) |
| C15—H15 | 0.95 | C45—H45 | 0.95 |
| C15—C16 | 1.390 (3) | C45—C46 | 1.380 (2) |
| | | | |
| H71—N1—C3 | 109.70 (10) | C15—C16—H16 | 119.61 |
| H71—N1—C4 | 107.37 (11) | C8—C21—C22 | 118.95 (12) |
| H71—N1—C7 | 106.90 (11) | C8—C21—C26 | 122.30 (13) |

| | | | |
|---|---|---|---|
| C3—N1—C4 | 112.27 (12) | C22—C21—C26 | 118.75 (14) |
| C3—N1—C7 | 110.59 (11) | C21—C22—H22 | 119.72 |
| C4—N1—C7 | 109.82 (10) | C21—C22—C23 | 120.56 (14) |
| C5—N2—C6 | 107.42 (10) | H22—C22—C23 | 119.72 |
| C5—N2—C8 | 110.41 (11) | C22—C23—H23 | 119.9 |
| C6—N2—C8 | 110.71 (11) | C22—C23—C24 | 120.20 (16) |
| H1a—C1—C2 | 116.52 | H23—C23—C24 | 119.9 |
| H1a—C1—C11 | 116.52 | C23—C24—H24 | 120.2 |
| C2—C1—C11 | 126.96 (14) | C23—C24—C25 | 119.60 (17) |
| C1—C2—H2 | 118.03 | H24—C24—C25 | 120.2 |
| C1—C2—C3 | 123.95 (13) | C24—C25—H25 | 119.64 |
| H2—C2—C3 | 118.03 | C24—C25—C26 | 120.72 (15) |
| N1—C3—C2 | 112.26 (13) | H25—C25—C26 | 119.64 |
| N1—C3—H3a | 109.47 | C21—C26—C25 | 120.16 (14) |
| N1—C3—H3b | 109.47 | C21—C26—H26 | 119.92 |
| C2—C3—H3a | 109.47 | C25—C26—H26 | 119.92 |
| C2—C3—H3b | 109.47 | C8—C31—C32 | 119.98 (11) |
| H3a—C3—H3b | 106.54 | C8—C31—C36 | 121.61 (13) |
| N1—C4—H4a | 109.47 | C32—C31—C36 | 118.41 (13) |
| N1—C4—H4b | 109.47 | C31—C32—H32 | 119.64 |
| N1—C4—C5 | 110.53 (12) | C31—C32—C33 | 120.73 (13) |
| H4a—C4—H4b | 108.39 | H32—C32—C33 | 119.64 |
| H4a—C4—C5 | 109.47 | C32—C33—H33 | 119.85 |
| H4b—C4—C5 | 109.47 | C32—C33—C34 | 120.30 (16) |
| N2—C5—C4 | 110.21 (12) | H33—C33—C34 | 119.85 |
| N2—C5—H5a | 109.47 | C33—C34—H34 | 120.18 |
| N2—C5—H5b | 109.47 | C33—C34—C35 | 119.65 (15) |
| C4—C5—H5a | 109.47 | H34—C34—C35 | 120.18 |
| C4—C5—H5b | 109.47 | C34—C35—H35 | 119.87 |
| H5a—C5—H5b | 108.72 | C34—C35—C36 | 120.25 (14) |
| N2—C6—H6a | 109.47 | H35—C35—C36 | 119.88 |
| N2—C6—H6b | 109.47 | C31—C36—C35 | 120.65 (15) |
| N2—C6—C7 | 110.93 (12) | C31—C36—H36 | 119.67 |
| H6a—C6—H6b | 107.97 | C35—C36—H36 | 119.68 |
| H6a—C6—C7 | 109.47 | C41—O1—H7 | 101.64 (10) |
| H6b—C6—C7 | 109.47 | C9—O7—H7 | 112.68 (16) |
| N1—C7—C6 | 110.95 (12) | O2—N3—O3 | 123.16 (15) |
| N1—C7—H7a | 109.47 | O2—N3—C46 | 118.72 (15) |
| N1—C7—H7b | 109.47 | O3—N3—C46 | 118.10 (13) |
| C6—C7—H7a | 109.47 | O4—N4—O5 | 122.91 (15) |
| C6—C7—H7b | 109.47 | O4—N4—C44 | 118.79 (14) |
| H7a—C7—H7b | 107.95 | O5—N4—C44 | 118.29 (16) |
| N2—C8—H8 | 108.39 | O6—C9—O7 | 122.64 (19) |
| N2—C8—C21 | 111.04 (11) | O6—C9—C42 | 122.44 (16) |
| N2—C8—C31 | 110.45 (11) | O7—C9—C42 | 114.91 (15) |
| H8—C8—C21 | 107.41 | O1—C41—C42 | 120.00 (15) |
| H8—C8—C31 | 108.03 | O1—C41—C46 | 125.00 (14) |
| C21—C8—C31 | 111.38 (10) | C42—C41—C46 | 115.00 (13) |

| | | | |
|---|---|---|---|
| C1—C11—C12 | 122.33 (15) | C9—C42—C41 | 121.64 (14) |
| C1—C11—C16 | 119.24 (13) | C9—C42—C43 | 116.80 (14) |
| C12—C11—C16 | 118.42 (15) | C41—C42—C43 | 121.55 (16) |
| C11—C12—H12 | 119.81 | C42—C43—H43 | 119.87 |
| C11—C12—C13 | 120.37 (17) | C42—C43—C44 | 120.27 (14) |
| H12—C12—C13 | 119.81 | H43—C43—C44 | 119.87 |
| C12—C13—H13 | 119.82 | N4—C44—C43 | 120.01 (14) |
| C12—C13—C14 | 120.37 (17) | N4—C44—C45 | 118.40 (15) |
| H13—C13—C14 | 119.82 | C43—C44—C45 | 121.59 (13) |
| C13—C14—H14 | 119.8 | C44—C45—H45 | 120.57 |
| C13—C14—C15 | 120.40 (18) | C44—C45—C46 | 118.86 (15) |
| H14—C14—C15 | 119.8 | H45—C45—C46 | 120.57 |
| C14—C15—H15 | 120.17 | N3—C46—C41 | 120.55 (13) |
| C14—C15—C16 | 119.7 (2) | N3—C46—C45 | 116.73 (14) |
| H15—C15—C16 | 120.17 | C41—C46—C45 | 122.71 (13) |
| C11—C16—C15 | 120.77 (16) | O1—H7—O7 | 148.96 (9) |
| C11—C16—H16 | 119.61 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H71···O1$^{i}$ | 0.9448 (11) | 1.9544 (11) | 2.8126 (15) | 150.01 (8) |
| N1—H71···O2$^{i}$ | 0.9448 (11) | 2.3020 (16) | 3.032 (2) | 133.62 (8) |
| C3—H3*a*···O6$^{ii}$ | 0.99 | 2.40 | 3.340 (2) | 159 |
| C5—H5*a*···C21 | 0.99 | 2.47 | 2.8777 (19) | 104 |
| C6—H6*b*···C31 | 0.99 | 2.50 | 2.8974 (19) | 104 |
| O7—H7···O1 | 0.9238 (15) | 1.6675 (13) | 2.505 (2) | 148.96 (9) |
| O7—H7···C41 | 0.9238 (15) | 2.2985 (16) | 2.827 (2) | 115.91 (9) |

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