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# Sodium 3,5-dinitrobenzoate

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**Key indicators**

Single-crystal X-ray study
*T* = 150 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.025
*wR* factor = 0.066
Data-to-parameter ratio = 7.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.



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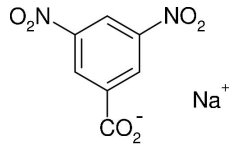
Sodium 3,5-dinitrobenzoate, $Na^+ \cdot C_7H_3N_2O_6^-$, was obtained by evaporation at room temperature of an aqueous solution of ethylenediammonium 3,5-dinitrobenzoate in sodium hydroxide. The structure is trigonal and the benzoate ion has twofold crystallographic symmetry.

## Comment

During work on crystallization of the salt ethylenediammonium 3,5-dinitrobenzoate, an aqueous solution of the salt at pH 12 was prepared and allowed to evaporate at room temperature, giving red prisms of sodium 3,5-dinitrobenzoate (NaDNB), (I). The crystal structure was not found in the Cambridge Structural Database (CSD, Version 5.25; Allen, 2002) and hence its structure was determined by single-crystal X-ray diffraction at 150 K.




(I)

The benzoate ion is on a twofold axis of symmetry, passing through the carboxylate group (Fig. 1).

## Experimental

3,5-Dinitrobenzoic acid (Aldrich, 99%) was dissolved in sodium hydroxide solution and a solution of ethylenediamine (Aldrich, 99%) was added. The solution was filtered and the pH recorded as 12.14. The solution pH was measured using an Accumet Basic AB15 pH

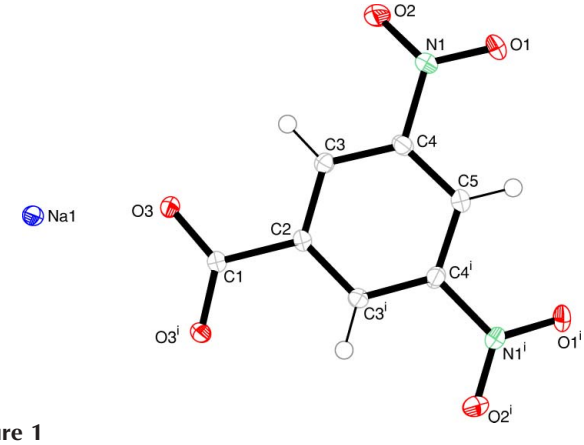



**Figure 1**
View of NaDNB, showing the whole benzoate anion. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $x-y, -y, -z+\frac{2}{3}$.]

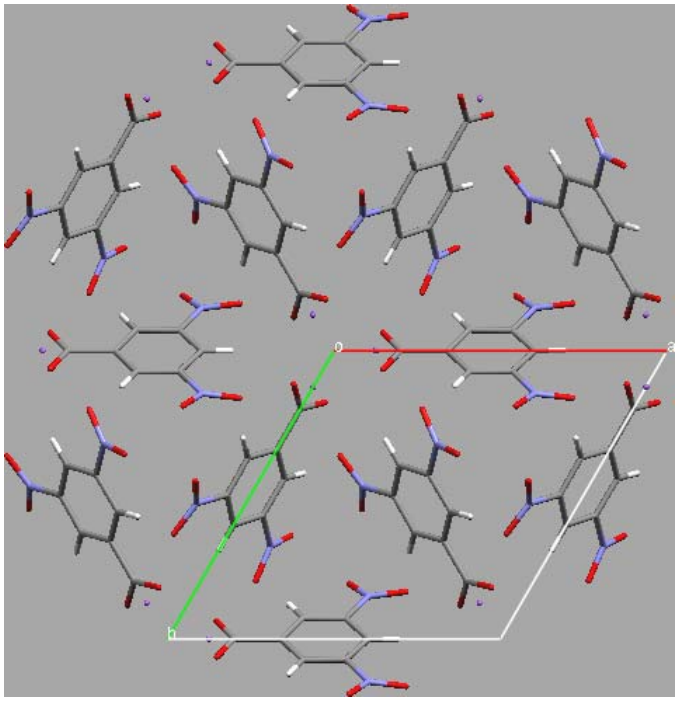


**Figure 2**
The packing of sodium 3,5-dinitrobenzoate, viewed along the *c* axis, showing the threefold symmetry.

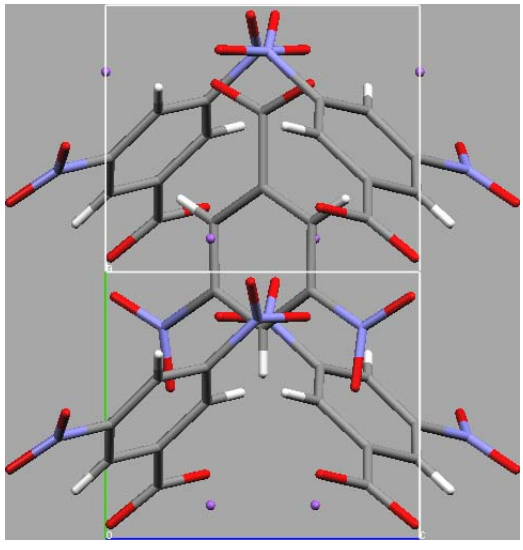


**Figure 3**
The twofold axis of symmetry perpendicular to the *c* axis.

meter with an Accumet glass calomel pH electrode. The solution was allowed to evaporate to dryness in air at room temperature. Crystals of ethylenediammonium 3,5-dinitrobenzoate, sodium hydroxide and red prisms of sodium 3,5-dinitrobenzoate formed.

*Crystal data*

$Na^+ \cdot C_7H_3N_2O_6^-$
$M_r$ = 234.1
Trigonal, $P3_121$
$a$ = 10.7701 (5) Å
$c$ = 6.3526 (2) Å
$V$ = 638.15 (5) Å$^3$
$Z$ = 3
$D_x$ = 1.828 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2522 reflections
$\theta$ = 1.0–27.5°
$\mu$ = 0.20 mm$^{-1}$
$T$ = 150 K
Prism, red
0.25 × 0.25 × 0.25 mm

*Data collection*

Nonius KappaCCD diffractometer
Thick-slice $\varphi$ and $\omega$ scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min}$ = 0.796, $T_{max}$ = 0.951
3498 measured reflections
554 independent reflections
537 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 27.5°
$h = -12 \rightarrow 13$
$k = -8 \rightarrow 13$
$l = -8 \rightarrow 8$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.066
$S$ = 1.09
554 reflections
76 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2 + 0.1582P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.14 (2)

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. The choice of space group $P3_121$ rather than $P3_221$ is arbitrary. All H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SORTAV* (Blessing, 1987,1989, *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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## References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Blessing, R. H. (1987). *Crystallogr. Rev.* **1**, 3–58.
Blessing, R. H. (1989). *J. Appl. Cryst.* **22**, 396–397.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

# supporting information

*Acta Cryst.* (2005). E**61**, m1131–m1132 [https://doi.org/10.1107/S1600536805014893]

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*Crystal data*

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$M_r = 234.1$
Trigonal, $P3_121$
Hall symbol: P 31 2"
$a = 10.7701$ (5) Å
$c = 6.3526$ (2) Å
$V = 638.15$ (5) Å$^3$
$Z = 3$
$F(000) = 354$
$D_x = 1.828$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2522 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.20$ mm$^{-1}$
$T = 150$ K
Prism, red
0.25 × 0.25 × 0.25 mm

*Data collection*

Nonius KappaCCD diffractometer
Radiation source: Enraf Nonius FR590
Graphite monochromator
CCD rotation images, thick slices scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.796$, $T_{max} = 0.951$
3498 measured reflections
554 independent reflections
537 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.8°$
$h = -12 \rightarrow 13$
$k = -8 \rightarrow 13$
$l = -8 \rightarrow 8$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.066$
$S = 1.09$
554 reflections
76 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2 + 0.1582P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.18$ e Å$^{-3}$
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.14 (2)

*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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.2002 (2) | 1 | −0.1667 | 0.0113 (5) |
| C2 | 0.3418 (2) | 1 | −0.1667 | 0.0122 (5) |
| C3 | 0.3701 (2) | 0.92732 (19) | −0.0104 (2) | 0.0131 (4) |
| H3 | 0.3033 | 0.8786 | 0.0952 | 0.016* |
| O3 | 0.11648 (14) | 0.93745 (13) | −0.01613 (17) | 0.0138 (3) |
| C4 | 0.4994 (2) | 0.9287 (2) | −0.0143 (2) | 0.0151 (4) |
| C5 | 0.6034 (2) | 1 | −0.1667 | 0.0155 (5) |
| H5 | 0.6897 | 1 | −0.1667 | 0.019* |
| N2 | 0.52972 (17) | 0.85204 (18) | 0.1513 (2) | 0.0186 (4) |
| Na1 | 0.87486 (9) | 0.87486 (9) | 0 | 0.0131 (3) |
| O1 | 0.63430 (15) | 0.83610 (16) | 0.1274 (2) | 0.0242 (4) |
| O2 | 0.45083 (19) | 0.8090 (2) | 0.3043 (2) | 0.0329 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0111 (8) | 0.0108 (11) | 0.0118 (10) | 0.0054 (5) | −0.0016 (4) | −0.0031 (8) |
| C2 | 0.0115 (9) | 0.0135 (11) | 0.0122 (10) | 0.0067 (6) | −0.0012 (4) | −0.0024 (9) |
| C3 | 0.0136 (9) | 0.0142 (9) | 0.0121 (8) | 0.0073 (7) | 0.0010 (6) | 0.0001 (6) |
| O3 | 0.0118 (6) | 0.0162 (7) | 0.0133 (6) | 0.0069 (5) | 0.0013 (4) | 0.0010 (5) |
| C4 | 0.0170 (8) | 0.0185 (9) | 0.0132 (8) | 0.0115 (7) | −0.0009 (6) | 0.0006 (7) |
| C5 | 0.0130 (9) | 0.0179 (13) | 0.0173 (11) | 0.0089 (6) | 0.0002 (5) | 0.0005 (9) |
| N2 | 0.0179 (8) | 0.0226 (9) | 0.0183 (7) | 0.0125 (7) | 0.0007 (6) | 0.0056 (6) |
| Na1 | 0.0131 (4) | 0.0131 (4) | 0.0128 (4) | 0.0064 (4) | −0.00068 (18) | 0.00068 (18) |
| O1 | 0.0170 (7) | 0.0323 (9) | 0.0300 (7) | 0.0174 (7) | 0.0027 (6) | 0.0104 (6) |
| O2 | 0.0356 (9) | 0.0548 (11) | 0.0228 (7) | 0.0335 (9) | 0.0132 (6) | 0.0209 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O3 | 1.2547 (16) | O3—Na1$^{ii}$ | 2.3416 (14) |
| C1—C2 | 1.525 (3) | C4—C5 | 1.386 (2) |
| C2—C3 | 1.389 (2) | C4—N2 | 1.471 (2) |
| C3—C4 | 1.386 (2) | C5—H5 | 0.93 |
| C3—H3 | 0.93 | N2—O2 | 1.220 (2) |
| O3—Na1$^{i}$ | 2.3083 (11) | N2—O1 | 1.231 (2) |
| | | | |
| O3$^{iii}$—C1—O3 | 126.5 (2) | O3$^{iv}$—Na1—O3$^{v}$ | 167.89 (8) |
| O3—C1—C2 | 116.77 (11) | O3$^{v}$—Na1—O3$^{vi}$ | 86.31 (5) |
| C3$^{iii}$—C2—C3 | 119.9 (2) | O3$^{v}$—Na1—O3$^{vii}$ | 102.24 (5) |
| C3—C2—C1 | 120.06 (11) | O3$^{vi}$—Na1—O3$^{vii}$ | 91.20 (7) |

| | | | |
|---|---|---|---|
| C4—C3—C2 | 118.90 (16) | O3$^{iv}$—Na1—O1 | 79.55 (5) |
| C4—C3—H3 | 120.5 | O3$^{v}$—Na1—O1 | 93.24 (5) |
| C2—C3—H3 | 120.5 | O3$^{vi}$—Na1—O1 | 82.64 (5) |
| C1—O3—Na1$^{i}$ | 131.53 (9) | O3$^{vii}$—Na1—O1 | 162.96 (5) |
| C1—O3—Na1$^{ii}$ | 125.81 (12) | O1$^{viii}$—Na1—O1 | 107.43 (8) |
| Na1$^{i}$—O3—Na1$^{ii}$ | 85.34 (5) | O3$^{iv}$—Na1—Na1$^{ix}$ | 47.77 (3) |
| C3—C4—C5 | 123.23 (16) | O3$^{v}$—Na1—Na1$^{ix}$ | 143.85 (5) |
| C3—C4—N2 | 119.02 (15) | O3$^{vi}$—Na1—Na1$^{ix}$ | 77.64 (3) |
| C5—C4—N2 | 117.74 (16) | O3$^{vii}$—Na1—Na1$^{ix}$ | 46.88 (4) |
| C4$^{iii}$—C5—C4 | 115.8 (2) | O1$^{viii}$—Na1—Na1$^{ix}$ | 108.62 (3) |
| C4—C5—H5 | 122.1 | O1—Na1—Na1$^{ix}$ | 116.10 (3) |
| O2—N2—O1 | 123.91 (16) | Na1$^{ix}$—Na1—Na1$^{x}$ | 100.20 (3) |
| O2—N2—C4 | 118.34 (14) | N2—O1—Na1 | 160.96 (12) |
| O1—N2—C4 | 117.74 (15) | | |

Symmetry codes: (i) $-y+1, x-y+1, z+1/3$; (ii) $x-1, y, z$; (iii) $x-y+1, -y+2, -z-1/3$; (iv) $-x+1, -x+y, -z+1/3$; (v) $-x+y, -x+1, z-1/3$; (vi) $y, x+1, -z$; (vii) $x+1, y, z$; (viii) $y, x, -z$; (ix) $-y+2, x-y+1, z+1/3$; (x) $-x+y+1, -x+2, z-1/3$.