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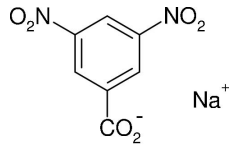
Sodium 3,5-dinitrobenzoate, $Na^+ \cdot C_7H_3N_2O_6^-$, was obtained by evaporation at room temperature of an aqueous solution of ethylenediammonium 3,5-dinitrobenzoate in sodium hydroxide. The structure is trigonal and the benzoate ion has twofold crystallographic symmetry.

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

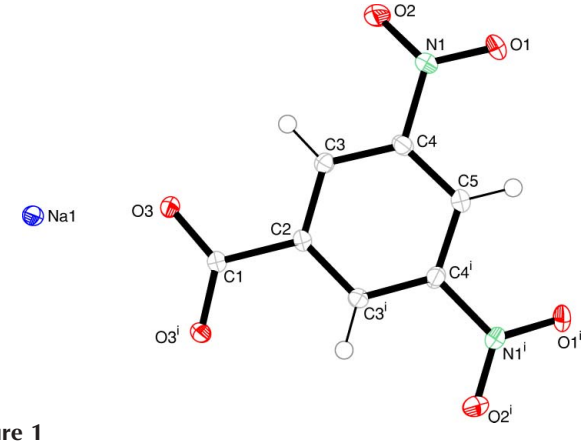


Figure 1
View of NaDNB, showing the whole benzoate anion. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $x-y, -y, -z+\frac{2}{3}$.]

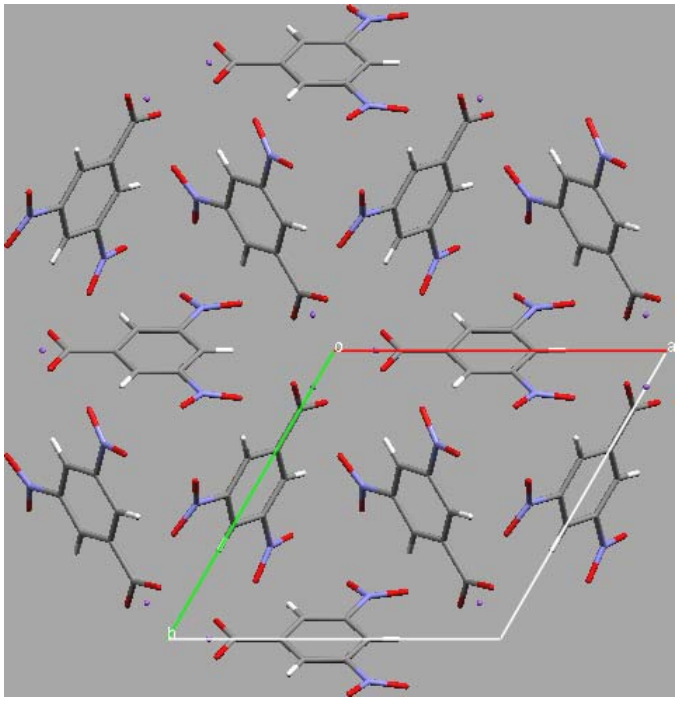


Figure 2
The packing of sodium 3,5-dinitrobenzoate, viewed along the *c* axis, showing the threefold symmetry.

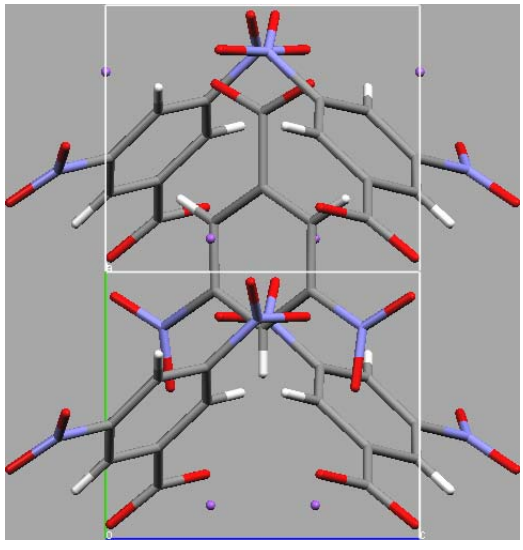


Figure 3
The twofold axis of symmetry perpendicular to the *c* axis.

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
θ = 1.0–27.5°
μ = 0.20 mm^{-1}
T = 150 K
Prism, red
0.25 × 0.25 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
Thick-slice φ and ω 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
θ_{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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