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Structure of Sodium *p*-Nitrophenolate Dihydrate

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Abstract. $[Na(C_6H_4NO_3)].2H_2O$, $M_r = 197.12$, orthorhombic, $Ima2$, $a = 6.892\,(1)$, $b = 19.692\,(2)$, $c = 6.439\,(1)$ Å, $V = 873.8\,(1)$ Å^3, $Z = 4$, $D_x = 1.498$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 15.1$ cm^{-1}, $F(000) = 408$, $T = 295$ K, $R = 0.066$ for 357 observed reflections. The *p*-nitrophenolate molecule, as well as the Na cation, is located on the crystallographic mirror plane. The Na cation is in contact with four O atoms, the distances being between 2.32 and 2.66 Å. The solvated water molecule is hydrogen bonded to the two O atoms of the NO_2 group.

Experimental. Crystals were grown as yellow square bipyramids from methanol solution. A well shaped crystal with dimensions of 0.50 × 0.50 × 0.80 mm was mounted on a Rigaku automated four-circle diffractometer. Unit-cell parameters were determined by a least-squares fit of 2θ values for 25 reflections in the range 45.7–69.8°. Intensities were measured by the θ–2θ-scan technique using Ni-filtered Cu $K\alpha$ radiation. The scan rate was 4° min^{-1} in 2θ and the scan width was $\Delta(2\theta) = (1.6 + 0.30\tan\theta)°$. Background intensities were measured for 5 s at the end of each scan. Four standard reflections (0,0,12, 228, 044, 400) were remeasured every 60 reflections, no significant loss of intensities was observed. 663 reflections were collected with 2θ up to 116° ($\sin\theta/\lambda = 0.550$ Å^{-1}) and an index range of $h = 0$ to 7, $k = 0$ to 21, $l = -7$ to 7. By averaging Friedel-pair reflections, 367 independent reflections were obtained ($R_{\rm int} = 0.010$). Corrections for Lorentz and polarization effects were applied, while neither absorption nor extinction corrections were carried out.

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) and refined by the block-diagonal least-squares procedure with program *HBLS*-V (Ashida, 1979). On the difference Fourier maps all the H atoms were found at the expected positions and were included in further refinements. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma(F_o)^2 + 0.010|F_o|^2]^{-1}$. At the final stage of the refinement, nine strong reflections (080, 170, 200, 220, 400, 011, 211, 202 and 222) affected by extinction were excluded. The number of observations per refined parameters was 357/91 = 3.92 and $S = 1.11$. The final R and wR were 0.066 and 0.088 for 357 observed reflections [$|F_o| \geq 3\sigma(|F_o|)$], respectively. $(\Delta/\sigma)_{\max}$ for the non-H atoms in the final refinement cycle was 0.24. The peaks in the final $\Delta\rho$ map were between 0.21 and

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq} (Å^2)*
Na	0.25	0.72822 (8)	0.4188 (5)	1.66
O(1)	0.25	0.32165 (14)	0.8812 (8)	1.63
O(2)	0.25	0.61597 (26)	0.5281 (14)	4.93
O(3)	0.25	0.63794 (18)	0.8585 (12)	3.99
N	0.25	0.5967 (3)	0.7135 (13)	3.25
C(1)	0.25	0.3862 (3)	0.8430 (11)	1.66
C(2)	0.25	0.4106 (4)	0.6343 (12)	2.72
C(3)	0.25	0.4816 (3)	0.5985 (12)	3.39
C(4)	0.25	0.5253 (3)	0.7544 (10)	2.06
C(5)	0.25	0.5027 (3)	0.9635 (13)	2.85
C(6)	0.25	0.4349 (4)	1.0033 (13)	2.92
O(*W*)	−0.0064 (4)	0.26254 (14)	0.1633 (6)	2.27

* As defined by Hamilton (1959).

Table 2. *Bond distances* (Å) *and angles* (°) *for non-*H *atoms and hydrogen-bond parameters* (Å, °) *with e.s.d.'s in parentheses*

O(1)—C(1)	1.295 (9)	O(2)—N	1.253 (12)
O(3)—N	1.238 (12)	N—C(4)	1.430 (11)
C(1)—C(2)	1.427 (10)	C(1)—C(6)	1.409 (11)
C(2)—C(3)	1.417 (11)	C(3)—C(4)	1.322 (10)
C(4)—C(5)	1.418 (11)	C(5)—C(6)	1.358 (12)
O(2)—N—O(3)	121.3 (9)	O(2)—N—C(4)	118.2 (8)
O(3)—N—C(4)	120.4 (8)	O(1)—C(1)—C(2)	120.6 (6)
O(1)—C(1)—C(6)	121.9 (7)	C(2)—C(1)—C(6)	117.4 (7)
C(1)—C(2)—C(3)	119.0 (7)	C(2)—C(3)—C(4)	121.2 (7)
N—C(4)—C(3)	120.0 (7)	N—C(4)—C(5)	118.9 (7)
C(3)—C(4)—C(5)	121.1 (7)	C(4)—C(5)—C(6)	119.2 (8)
C(1)—C(6)—C(5)	122.0 (8)		

O—H···O	O···O	O—H···O
O(*W*)—H(*W*1^{i})···O(1^{ii})	2.789 (6)	173 (7)
O(*W*)—H(*W*2^{i})···O(1^{iii})	2.800 (6)	178 (9)

Symmetry code: (i) x, y, z; (ii) $x, y, -1+z$; (iii) $x, \frac{1}{2}-y, -\frac{1}{2}+z$.

-0.18 e Å^{-3}. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final atomic parameters are listed in Table 1.* The molecular structure with atomic numbering system and the crystal-packing diagram are depicted in Figs. 1 and 2, respectively. Bond distances and angles are presented in Table 2. All the computations were performed on an ACOS 930 computer at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

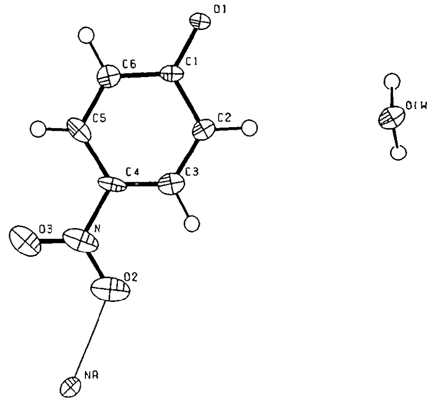

Fig. 1. *ORTEP*II drawing (Johnson, 1976) of the molecular structure together with atomic numbering system. Non-H atoms are represented by thermal ellipsoids with 50% probability levels, whereas H atoms are drawn by a sphere with $B = 1.0$ Å^2.

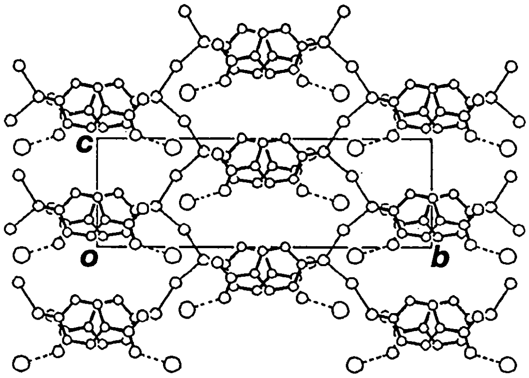

Fig. 2. *ORTEP*II drawing (Johnson, 1976) of ther crystal packing diagram as viewed along the *a* axis. Na, O, N and C atoms are represented by spheres with $B = 8$, 6, 5 and 4 Å^2, respectively, whereas H atoms are omitted for clarity. The hydrogen bonds are drawn by thin solid lines, whereas the contacts between Na and O(2) are shown by dotted lines.

Related literature. The Na cation strongly interacts with the O(2) atom of the NO_2 group [2.320 (9) Å] and is also in contact with O(3) and the two O(*W*) atoms with distances of 2.664 (8), 2.358 (5) and 2.398 (5) Å, respectively. The O(*W*) atom of the solvated water is hydrogen bonded to the O(1) atoms of *p*-nitrophenolate, as presented in Table 2. These hydrogen bonds extend to form a network along the direction of the crystallographic *c* axis. The structure of 1-chloro-3-nitrobenzene has been reported (Sharma, Paulus, Weiden & Weiss, 1985).

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* Lists of anisotropic temperature factors, atomic parameters for H atoms, structure factors and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54597 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0531]