# Crystal structure of 1-adamantylammonium 4-nitrophenolate hydrate, $(C_{10}H_{15}NH_3)(OC_6H_4NO_2) \cdot H_2O$

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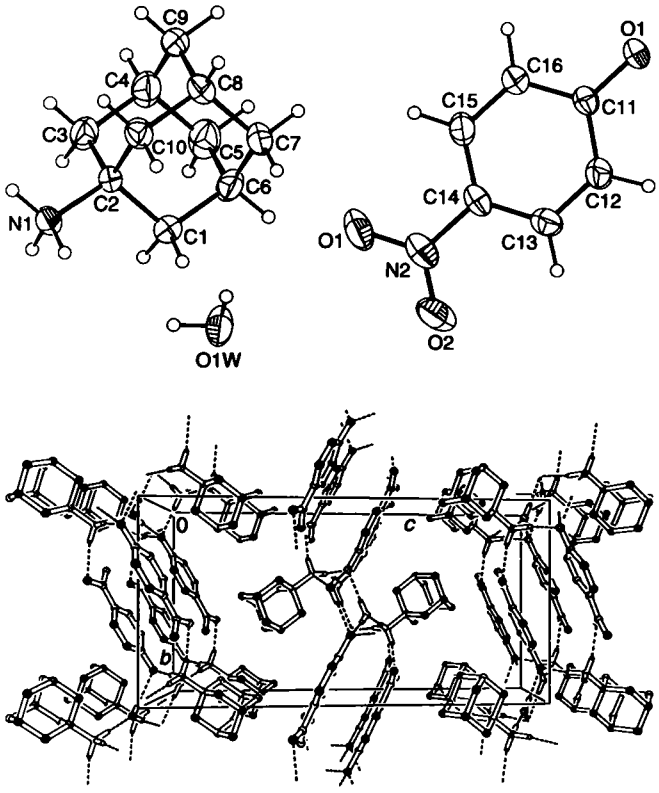


**Abstract**

$C_{16}H_{24}N_2O_4$, monoclinic, $P12_1/n1$ (no. 14),
$a = 6.433(4)$ Å, $b = 11.366(2)$ Å, $c = 22.406(3)$ Å,
$\beta = 94.77(4)°$, $V = 1632.6$ Å$^3$, $Z = 4$,
$R_{gt}(F) = 0.057$, $wR_{ref}(F^2) = 0.144$, $T = 293$ K.

**Source of material**

13.9 g (0.1 mol) 4-nitrophenol and 1.51 g (0.1 mol) 1-adamantanamine were weighted, respectively, and mixed with 20 mL ethanol and 10 mL water together, and then heated until the solid had dissolved completely. After cooling gradually and standing still at room temperature for a few days, the white crystalline solid suitable for X-ray diffraction were obtained.

**Experimental details**

Although all H atom parameters converge if freely refined the *X*—H bonds were restrained to ideal values due to the small $N(hkl)_{gt}/N(param)_{refined}$ ratios of about five and seven, respectively ($d_{C—H} = 0.97$ Å, $d_{C_{aryl}—H} = 0.93$ Å, $d_{N—H} = 0.89$ Å, $d_{O—H} = 0.84$ Å).

**Discussion**

In recent years, much attention has been paid to weak intra- and inter-molecular interactions, which play an important role in the field of chemistry and biochemistry [1]. As part of the investigation on the complexes formed by nitrophenol, which could exhibit non-linear optics [2], the crystal structure by *para*-nitrophenol, 1-adamantanamine and water has been determined.

One asymmetric unit comprises one 4-nitrophenolate, one 1-adamantylammonium and one water molecule (figure, top). The 4-nitrophenolato ion is planar, and the conformation of the 1-adamantyl unit is the same as that of hexamethylenetetramine, with the corresponding bond lengths: *d*(C1—C2) = 1.512(4) Å, *d*(C1—C6) = 1.526(4) Å, *d*(C2—N1) = 1.498(3) Å, *d*(C2—C10) = 1.514(4) Å, *d*(C2—C3) = 1.517(4) Å, *d*(C3—C4) = 1.528(4) Å, *d*(C4—C9) = 1.511(6) Å, *d*(C4—C5) = 1.532(5) Å, *d*(C5—C6) = 1.519(5) Å, *d*(C6—C7) = 1.519(5) Å, *d*(C7—C8) = 1.526(4) Å, *d*(C8—C10) = 1.524(4) Å, *d*(C8—C9) = 1.537(5) Å.

Five strong hydrogen bonds (O–H···O, N–H···O) are involved in the complex structure to stabilize the crystal packing through layer stacking along the *b*-axis (figure, bottom), these are *d*[N1···O1($x$,$y$,$-1+z$)] = 2.746 Å, *d*(H1N···O1) = 1.867 Å, ∠N1–H1N···O1 = 168.6°; *d*[N1–O3($2-x$,$1-y$,$1-z$)] = 2.953 Å, *d*(H2N···O3) = 2.081 Å, ∠N1–H2N···O3 = 166.4°; *d*[N1–O1W($1+x$,$y$,$z$)] = 2.746 Å, *d*(H3N···O1W) = 1.859 Å, ∠N1–H3N···O1W = 174.1°; *d*[O1W–O1($1-x$,$2-y$,$1-z$)] = 2.774 Å, *d*(H1WA···O1) = 1.943 Å, ∠O1W–H1WA···O1 = 170.0°; *d*[O1W–O1($x$,$y$,$-1+z$)] = 2.736 Å, *d*(H1WB···O1) = 1.904 Å, ∠O1W–H1WB···O1 = 170.2°.

Additionally, four weak C–H···O and C–H···N interactions are also present in the crystal: *d*[C3–O1W($1+x$,$y$,$z$)] = 3.738 Å, *d*(H3A···O1W) = 3.090 Å, ∠C3–H3A···O1W = 125.5°; *d*[C9–O2($x+½$,$-y+¾$,$z-½$)] = 3.724 Å, *d*(H9A···O2) = 3.066 Å, ∠C9–H9A···O2 = 126.3°; *d*[C10–O1W($1+x$,$y$,$z$)] = 3.856 Å, *d*(H10A···O1W) = 3.086 Å, ∠C10–H10A···O1W = 137.3°; *d*[C4–N2($x+½$,$-y+¾$,$z-½$)] = 3.644 Å, *d*(H4···N2) = 2.823 Å, ∠C4–H4···N2 = 141.8°.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colorless prism, size 0.40 x 0.60 x 0.60 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.90 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker P4, $\theta$-$2\theta$ |
| $2\theta_{max}$: | 50° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 3955, 2808 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1521 |
| $N(param)_{refined}$: | 295 |
| Programs: | SHELXTL [3], PLATON [4] |

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**Table 2.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1A) | 4*e* | 0.603(5) | 0.892(2) | 0.144(1) | 0.058(8) |
| H(1B) | 4*e* | 0.759(6) | 0.813(3) | 0.189(2) | 0.09(1) |
| H(3A) | 4*e* | 1.217(5) | 0.959(3) | 0.175(1) | 0.062(9) |
| H(3B) | 4*e* | 1.136(5) | 0.850(3) | 0.209(1) | 0.08(1) |
| H(4) | 4*e* | 1.198(6) | 1.015(3) | 0.275(2) | 0.09(1) |
| H(5A) | 4*e* | 0.839(6) | 1.036(4) | 0.306(2) | 0.12(2) |
| H(5B) | 4*e* | 0.879(6) | 0.891(4) | 0.290(2) | 0.10(2) |
| H(6) | 4*e* | 0.555(5) | 0.952(3) | 0.244(1) | 0.07(1) |
| H(7A) | 4*e* | 0.528(5) | 1.100(3) | 0.175(1) | 0.065(9) |
| H(7B) | 4*e* | 0.631(6) | 1.149(3) | 0.239(2) | 0.10(1) |
| H(8) | 4*e* | 0.824(5) | 1.215(3) | 0.161(1) | 0.08(1) |
| H(9A) | 4*e* | 1.143(6) | 1.167(3) | 0.202(2) | 0.09(1) |

**Table 2.** Continued.

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(9B) | 4*e* | 1.006(5) | 1.194(3) | 0.257(1) | 0.08(1) |
| H(10A) | 4*e* | 1.009(5) | 1.086(3) | 0.108(1) | 0.058(9) |
| H(10B) | 4*e* | 0.759(5) | 1.058(3) | 0.094(1) | 0.07(1) |
| H(1N) | 4*e* | 0.836(5) | 0.858(3) | 0.064(1) | 0.08(1) |
| H(2N) | 4*e* | 1.082(5) | 0.881(3) | 0.080(1) | 0.07(1) |
| H(3N) | 4*e* | 0.976(5) | 0.782(3) | 0.111(2) | 0.08(1) |
| H(12) | 4*e* | 0.391(5) | 0.691(3) | 0.968(1) | 0.07(1) |
| H(13) | 4*e* | 0.459(5) | 0.506(3) | 0.925(1) | 0.07(1) |
| H(15) | 4*e* | 1.072(6) | 0.588(3) | 0.927(2) | 0.09(1) |
| H(16) | 4*e* | 1.007(5) | 0.766(3) | 0.977(1) | 0.07(1) |
| H(1WA) | 4*e* | 0.311(7) | 1.001(4) | 0.028(2) | 0.12(2) |
| H(1WB) | 4*e* | 0.408(5) | 0.899(3) | 0.031(1) | 0.07(1) |

**Table 3.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| C(1) | 4*e* | 0.7240(5) | 0.8930(3) | 0.1767(2) | 0.046(2) | 0.053(2) | 0.056(2) | −0.003(2) | 0.006(2) | −0.006(2) |
| C(2) | 4*e* | 0.9192(4) | 0.9346(2) | 0.1492(1) | 0.040(2) | 0.040(2) | 0.052(2) | 0.001(1) | 0.009(1) | −0.005(1) |
| C(3) | 4*e* | 1.1056(5) | 0.9299(4) | 0.1953(2) | 0.042(2) | 0.077(3) | 0.066(2) | 0.007(2) | 0.001(2) | −0.010(2) |
| C(4) | 4*e* | 1.0650(6) | 1.0121(4) | 0.2470(2) | 0.050(2) | 0.114(4) | 0.074(2) | 0.005(2) | −0.001(2) | −0.039(2) |
| C(5) | 4*e* | 0.8697(6) | 0.9711(5) | 0.2754(2) | 0.070(3) | 0.114(4) | 0.057(2) | 0.006(3) | 0.008(2) | −0.017(3) |
| C(6) | 4*e* | 0.6847(5) | 0.9745(3) | 0.2285(2) | 0.045(2) | 0.085(3) | 0.065(2) | −0.007(2) | 0.020(2) | −0.014(2) |
| C(7) | 4*e* | 0.6513(6) | 1.0994(3) | 0.2051(2) | 0.052(2) | 0.073(3) | 0.086(3) | 0.009(2) | 0.011(2) | −0.032(2) |
| C(8) | 4*e* | 0.8464(6) | 1.1403(3) | 0.1771(2) | 0.068(3) | 0.045(2) | 0.107(3) | −0.001(2) | 0.028(2) | −0.019(2) |
| C(9) | 4*e* | 1.0352(6) | 1.1363(4) | 0.2244(2) | 0.059(3) | 0.097(4) | 0.118(3) | −0.022(2) | 0.032(3) | −0.067(3) |
| C(10) | 4*e* | 0.8868(6) | 1.0586(3) | 0.1252(2) | 0.062(2) | 0.042(2) | 0.078(2) | 0.004(2) | 0.021(2) | 0.003(2) |
| N(1) | 4*e* | 0.9616(5) | 0.8560(3) | 0.0979(1) | 0.053(2) | 0.043(2) | 0.055(2) | 0.005(1) | 0.011(2) | −0.005(1) |
| C(11) | 4*e* | 0.6927(4) | 0.7435(2) | 0.9767(1) | 0.052(2) | 0.038(2) | 0.049(2) | 0.008(2) | 0.006(1) | 0.001(1) |
| C(12) | 4*e* | 0.5310(5) | 0.6642(3) | 0.9599(2) | 0.047(2) | 0.059(2) | 0.074(2) | 0.010(2) | 0.001(2) | −0.013(2) |
| C(13) | 4*e* | 0.5682(6) | 0.5594(3) | 0.9337(2) | 0.062(2) | 0.054(2) | 0.070(2) | −0.004(2) | −0.003(2) | −0.017(2) |
| C(14) | 4*e* | 0.7692(5) | 0.5298(3) | 0.9216(1) | 0.068(2) | 0.040(2) | 0.047(2) | 0.013(2) | 0.006(2) | −0.002(1) |
| C(15) | 4*e* | 0.9326(6) | 0.6064(3) | 0.9363(2) | 0.059(2) | 0.057(2) | 0.069(2) | 0.010(2) | 0.021(2) | −0.005(2) |
| C(16) | 4*e* | 0.8951(5) | 0.7107(3) | 0.9637(2) | 0.057(2) | 0.047(2) | 0.077(2) | −0.001(2) | 0.018(2) | −0.011(2) |
| N(2) | 4*e* | 0.8109(6) | 0.4192(3) | 0.8952(1) | 0.093(2) | 0.047(2) | 0.057(2) | 0.014(2) | 0.009(2) | −0.002(1) |
| O(1) | 4*e* | 0.6597(3) | 0.8419(2) | 1.00431(9) | 0.057(1) | 0.042(1) | 0.072(1) | 0.009(1) | 0.010(1) | −0.010(1) |
| O(2) | 4*e* | 0.6714(5) | 0.3449(2) | 0.8885(1) | 0.113(2) | 0.055(2) | 0.105(2) | −0.001(2) | 0.001(2) | −0.023(2) |
| O(3) | 4*e* | 0.9890(5) | 0.3991(2) | 0.8797(1) | 0.110(2) | 0.060(2) | 0.096(2) | 0.023(2) | 0.042(2) | −0.008(1) |
| O(1W) | 4*e* | 0.3003(4) | 0.9343(3) | 0.0427(1) | 0.072(2) | 0.066(2) | 0.111(2) | 0.018(2) | 0.048(2) | 0.017(2) |

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