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2,3,5,6-Tetramethylpyrazine–*p*-nitrophenol (1/2)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.077
wR factor = 0.170
Data-to-parameter ratio = 13.1

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

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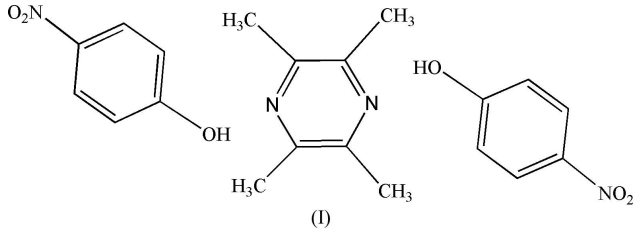
In the title complex, $C_8H_{12}N_2{\cdot}2C_6H_5NO_3$, one 2,3,5,6-tetramethylpyrazine and two *p*-nitrophenol molecules form a centrosymmetric unit. Intermolecular O—H···N and weak C—H···O hydrogen bonds are observed in the structure, leading to the formation of a two-dimensional network.

Comment

2,3,5,6-Tetramethylpyrazine (TMP), which possesses anti-ulcer and antiplatelet activities, is a biologically active ingredient isolated from the traditional herbal medicine *Ligusticum chuanxiong Hort*, widely used in China for the clinical treatment of nephrosis and ischemic cerebrovascular disease, due to its function of anticoagulation and angiectasis (Peng *et al.*, 2004). It also has a protective effect against ischemic neuronal damage in the hippocampus (Su *et al.*, 2004). *p*-Nitrophenol (PNP), which is commonly used as a pesticide in agriculture, is a precursor of pharmaceuticals such as acetaminophen and 4-aminosalicylic acid (Kam *et al.*, 2005). In the past, some supramolecular compounds were synthesized with either TMP (Tian & Yang, 1993; Smyth *et al.*, 1996; Dong *et al.*, 2003) or PNP (Paternostre *et al.*, 1998; Ng *et al.*, 2001). We report here the structure of the title compound, (I), containing both TMP and PNP.



(I)

In (I), one TMP molecule links two PNP molecules *via* O—H···N hydrogen-bond interactions to form a centrosymmetric unit (Fig. 1). The N2—C8, N2—C9, C9—C8^{i} distances [1.339 (3)–1.402 (3) Å; symmetry code: (i) $1-x, -y, 1-z$] are between double- and single-bond lengths, indicating

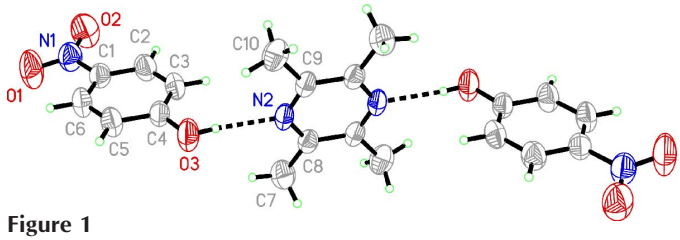


Figure 1
The structure of (I), with the atom numbering, showing displacement ellipsoids at the 50% probability level. Unlabeled atoms are related to labeled atoms by $1-x, -y, 1-z$.

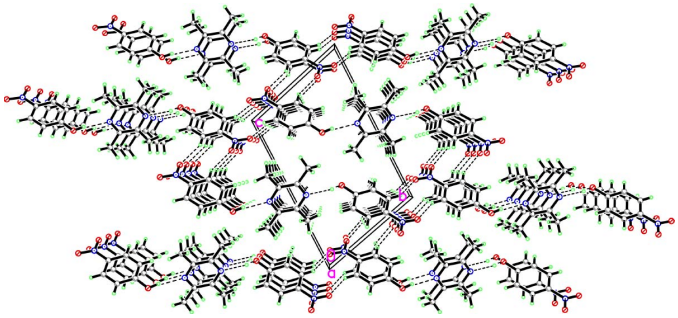


Figure 2
The two-dimensional network formed by hydrogen-bond interactions, which are shown as dashed lines.

electron delocalization in the pyrazine ring (Table 1). The dihedral angle between the pyrazine ring of TMP and the benzene ring of PNP is 82.38 (9)°. The weak intermolecular C—H···O hydrogen bonds (Table 2) link the centrosymmetric units into a two-dimensional network (Fig. 2).

Experimental

The title compound was obtained by the hydrothermal method from a mixture of 2,3,5,6-tetramethylpyrazine (2 mmol, 0.27 g) and *p*-nitrophenol (2 mmol, 0.28 g) in a 30 ml Teflon-lined stainless steel reactor. The solution was heated to 412 K for 3 d. On completion of the reaction, the system was cooled slowly to room temperature and the colorless block-shaped crystals which formed were collected.

Crystal data

$C_8H_{12}N_2{\cdot}2C_6H_5NO_3$
M_r = 414.42
Triclinic, $P\overline{1}$
a = 4.7521 (2) Å
b = 8.8700 (4) Å
c = 13.1941 (6) Å
α = 73.916 (2)°
β = 81.771 (2)°
γ = 74.706 (2)°
V = 513.98 (4) Å^3
Z = 1
D_x = 1.339 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1276 reflections
θ = 2.5–25.2°
μ = 0.10 mm^{-1}
T = 298 (2) K
Block, colorless
0.48 × 0.35 × 0.15 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.953, T_{max} = 0.985
2747 measured reflections
1825 independent reflections
1720 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 25.2°
$h = -5 \rightarrow 5$
$k = -8 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.170
S = 1.27
1825 reflections
139 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.2196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.225 (3)	N2—C9	1.340 (3)
O2—N1	1.219 (3)	C8—C9^{i}	1.402 (3)
N1—C1	1.464 (4)	C9—C8^{i}	1.402 (3)
N2—C8	1.339 (3)		
C4—O3—H3	109.5	O1—N1—C1	118.5 (3)
O2—N1—O1	122.7 (3)	C8—N2—C9	120.3 (2)
O2—N1—C1	118.8 (3)		

Symmetry code: (i) $1-x, -y, 1-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N2	0.82	2.01	2.818 (3)	170
C6—H6···O1^{ii}	0.93	2.57	3.335 (4)	140
C2—H2···O2^{iii}	0.93	2.52	3.380 (4)	154

Symmetry codes: (ii) $-x, 2-y, -z$; (iii) $2-x, 1-y, -z$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with Csp^2—H = 0.93 Å with U_{iso} = 1.2U_{eq}(C), Csp^3—H = 0.96 Å with U_{iso} = 1.5U_{eq}(C) and O—H = 0.82 Å with U_{iso} = 1.2U_{eq}(O).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002); software used to prepare material for publication: *XP* in *SHELXTL* (Bruker, 2002).

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