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2,4,6-Trimethylanilinium chloride

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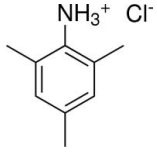
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.138; data-to-parameter ratio = 20.4.

The title compound, $C_9H_{14}N^+\cdot Cl^-$, is a hydrochloric acid salt of 2,4,6-trimethylaniline. In the crystal structure, all the hydrogen-bond donors and acceptors are involved in hydrogen bonds. The packing can be described as columns, two ion-pairs wide, propagating along the a axis. The columns are formed through N—H···Cl hydrogen bonds linking pairs of cations and anions around centers of symmetry and further connecting these pairs in the [100] direction. In addition, the aromatic rings on each side of the columns are stacked above each other, indicating π–π stacking (the distance between aromatic rings is 4.811 Å).

Related literature

For related literature, see: Ting *et al.* (1990).



Experimental

Crystal data

$C_9H_{14}N^+\cdot Cl^-$
M_r = 171.66
Monoclinic, $P2_1/c$
a = 4.811 (1) Å
b = 15.373 (3) Å
c = 12.509 (2) Å
β = 90.99 (3)°
V = 925.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 90.0 (2) K
0.40 × 0.13 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.873, T_{max} = 0.990
4000 measured reflections
2121 independent reflections
1453 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.04
2121 reflections
104 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1	0.91	2.18	3.081 (2)	172
N1—H1B···Cl1^{i}	0.91	2.33	3.181 (2)	155
N1—H1C···Cl1^{ii}	0.91	2.36	3.146 (2)	145

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $x+1, y, z$.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local programs.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2132).

References

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supplementary materials

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2,4,6-Trimethylanilinium chloride

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Comment

The title compound, (I), was a by-product during our attempts to synthesize 2-[(2,4,6-trimethylphenyl)amino]-3-pyridine-carboxylic acid, a potential anti-inflammatory compound (Ting *et al.*, 1990).

The asymmetric unit of the crystal I (Fig. 1) consists of a 2,4,6-trimethylbenzenaminium cation and a chloride anion·In the crystal of I, all the available hydrogen bonding donors and acceptors are involved in the three-dimensional hydrogen bonding network. The molecules in the crystal form columns with a width of two molecules along the *a* axis through N—H···Cl hydrogen bonds (Table 1). The N—H···Cl hydrogen bonds link pairs of molecules around a center of symmetry and further connect these pairs of molecules in the [1 0 0] direction. Moreover, the aromatic rings on each side of the columns are stacked face-to-face, suggesting π-π stacking may provide additional stablitity to the crystal structure (Fig. 2).

Experimental

Pyridine (3.78 g, 47.8 mmol) was introduced to a round bottom flask containing 2-chloronicotinic acid (7.32 g, 46 mmol) and 2,4,6-trimethylaniline (6.55 g, 48.5 mmol), followed by addition of *p*-toluenesulfonic acid (1.2 g, 6.98 mmol) dissovled in 20 ml of water. The solution was then refluxed overnight. After workup, both the desired product, 2-[(2,4,6-trimethylphenyl)amino]-3-pyridinecarboxylic acid, and some by-products, including the title compound, were obtained. Crystals of the title compound were grown from methanol solution by slow evaporation.

Figures

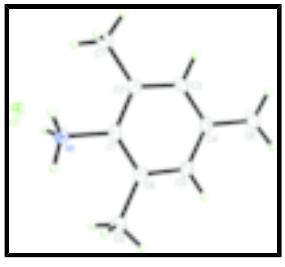

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

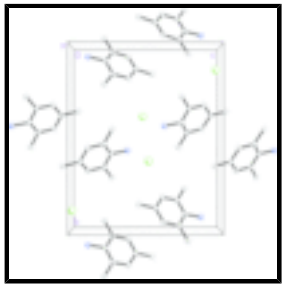

Fig. 2. A packing diagram of (I) along *a* axis.

2,4,6-Trimethylanilinium chloride

Crystal data

$C_9H_{14}N^+\cdot Cl^-$	F_{000} = 368
M_r = 171.66	D_x = 1.233 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 4.811 (1) Å	Cell parameters from 2207 reflections
b = 15.373 (3) Å	θ = 1.0–27.5º
c = 12.509 (2) Å	μ = 0.35 mm^{-1}
β = 90.99 (3)º	T = 90.0 (2) K
V = 925.0 (3) Å^3	Thick needle, colorless
Z = 4	0.40 × 0.13 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer	2121 independent reflections
Radiation source: fine-focus sealed tube	1453 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
Detector resolution: 18 pixels mm^{-1}	θ_{max} = 27.5º
T = 90.0(2) K	θ_{min} = 2.1º
ω scans at fixed χ = 55°	h = −6→6
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	k = −19→19
T_{min} = 0.873, T_{max} = 0.990	l = −16→16
4000 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.138	$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
2121 reflections	$\Delta\rho_{max}$ = 0.64 e Å^{-3}
104 parameters	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6201 (5)	0.08520 (15)	0.23416 (18)	0.0171 (5)
C2	0.4224 (5)	0.03061 (15)	0.27942 (17)	0.0175 (5)
C3	0.3333 (5)	0.05229 (15)	0.38169 (17)	0.0199 (5)
H3	0.2016	0.0158	0.4153	0.024*
C4	0.4299 (5)	0.12513 (14)	0.43603 (18)	0.0181 (5)
C5	0.6277 (5)	0.17733 (15)	0.38738 (17)	0.0179 (5)
H5	0.6980	0.2268	0.4244	0.021*
C6	0.7249 (4)	0.15857 (15)	0.28554 (18)	0.0161 (5)
C7	0.3084 (5)	−0.04773 (16)	0.22143 (19)	0.0229 (6)
H7A	0.4541	−0.0920	0.2161	0.034*
H7B	0.2455	−0.0307	0.1495	0.034*
H7C	0.1515	−0.0715	0.2609	0.034*
C8	0.3174 (5)	0.14835 (17)	0.54347 (19)	0.0249 (6)
H8A	0.4618	0.1780	0.5860	0.037*
H8B	0.2601	0.0953	0.5805	0.037*
H8C	0.1568	0.1870	0.5341	0.037*
C9	0.9377 (5)	0.21750 (15)	0.23440 (19)	0.0210 (6)
H9A	1.1011	0.1832	0.2150	0.031*
H9B	0.9933	0.2631	0.2851	0.031*
H9C	0.8561	0.2442	0.1700	0.031*
N1	0.7035 (4)	0.06625 (12)	0.12322 (13)	0.0173 (5)
H1A	0.5682	0.0848	0.0769	0.026*
H1B	0.7285	0.0079	0.1154	0.026*
H1C	0.8653	0.0944	0.1092	0.026*
Cl1	0.20588 (11)	0.12054 (4)	−0.02006 (4)	0.0188 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0180 (12)	0.0185 (12)	0.0147 (11)	0.0054 (10)	0.0009 (9)	−0.0003 (10)
C2	0.0176 (12)	0.0165 (12)	0.0184 (12)	0.0023 (10)	0.0002 (9)	0.0019 (10)
C3	0.0255 (13)	0.0158 (12)	0.0186 (12)	0.0018 (10)	0.0037 (10)	0.0049 (10)

C4	0.0230 (13)	0.0167 (12)	0.0146 (11)	0.0066 (10)	−0.0002 (9)	0.0012 (10)
C5	0.0225 (13)	0.0152 (12)	0.0158 (12)	0.0032 (10)	−0.0026 (10)	−0.0015 (9)
C6	0.0159 (12)	0.0142 (12)	0.0182 (12)	0.0041 (10)	−0.0014 (9)	−0.0005 (10)
C7	0.0243 (13)	0.0199 (13)	0.0246 (13)	−0.0044 (11)	0.0045 (10)	−0.0003 (11)
C8	0.0363 (16)	0.0207 (13)	0.0179 (12)	0.0035 (11)	0.0053 (11)	−0.0005 (10)
C9	0.0224 (13)	0.0180 (13)	0.0226 (13)	0.0007 (10)	0.0033 (10)	−0.0020 (11)
N1	0.0185 (10)	0.0160 (10)	0.0174 (10)	−0.0014 (8)	0.0021 (8)	−0.0015 (8)
Cl1	0.0212 (3)	0.0177 (3)	0.0177 (3)	−0.0002 (2)	0.0026 (2)	−0.0009 (2)

Geometric parameters (Å, °)

C1—C6	1.389 (3)	C7—H7A	0.9800
C1—C2	1.396 (3)	C7—H7B	0.9800
C1—N1	1.480 (3)	C7—H7C	0.9800
C2—C3	1.397 (3)	C8—H8A	0.9800
C2—C7	1.504 (3)	C8—H8B	0.9800
C3—C4	1.386 (3)	C8—H8C	0.9800
C3—H3	0.9500	C9—H9A	0.9800
C4—C5	1.393 (3)	C9—H9B	0.9800
C4—C8	1.501 (3)	C9—H9C	0.9800
C5—C6	1.395 (3)	N1—H1A	0.9100
C5—H5	0.9500	N1—H1B	0.9100
C6—C9	1.517 (3)	N1—H1C	0.9100
C6—C1—C2	123.0 (2)	C2—C7—H7C	109.5
C6—C1—N1	119.44 (19)	H7A—C7—H7C	109.5
C2—C1—N1	117.4 (2)	H7B—C7—H7C	109.5
C1—C2—C3	116.8 (2)	C4—C8—H8A	109.5
C1—C2—C7	122.06 (19)	C4—C8—H8B	109.5
C3—C2—C7	121.2 (2)	H8A—C8—H8B	109.5
C4—C3—C2	122.4 (2)	C4—C8—H8C	109.5
C4—C3—H3	118.8	H8A—C8—H8C	109.5
C2—C3—H3	118.8	H8B—C8—H8C	109.5
C3—C4—C5	118.5 (2)	C6—C9—H9A	109.5
C3—C4—C8	120.6 (2)	C6—C9—H9B	109.5
C5—C4—C8	120.9 (2)	H9A—C9—H9B	109.5
C4—C5—C6	121.5 (2)	C6—C9—H9C	109.5
C4—C5—H5	119.3	H9A—C9—H9C	109.5
C6—C5—H5	119.3	H9B—C9—H9C	109.5
C1—C6—C5	117.8 (2)	C1—N1—H1A	109.5
C1—C6—C9	122.2 (2)	C1—N1—H1B	109.5
C5—C6—C9	120.1 (2)	H1A—N1—H1B	109.5
C2—C7—H7A	109.5	C1—N1—H1C	109.5
C2—C7—H7B	109.5	H1A—N1—H1C	109.5
H7A—C7—H7B	109.5	H1B—N1—H1C	109.5
C6—C1—C2—C3	−0.8 (3)	C3—C4—C5—C6	1.1 (3)
N1—C1—C2—C3	−176.3 (2)	C8—C4—C5—C6	−177.2 (2)
C6—C1—C2—C7	179.0 (2)	C2—C1—C6—C5	0.5 (3)
N1—C1—C2—C7	3.5 (3)	N1—C1—C6—C5	175.93 (19)
C1—C2—C3—C4	1.2 (3)	C2—C1—C6—C9	−179.4 (2)

C7—C2—C3—C4	−178.5 (2)	N1—C1—C6—C9	−4.0 (3)
C2—C3—C4—C5	−1.4 (3)	C4—C5—C6—C1	−0.7 (3)
C2—C3—C4—C8	176.9 (2)	C4—C5—C6—C9	179.2 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl1	0.91	2.18	3.081 (2)	172
N1—H1B···Cl1^{i}	0.91	2.33	3.181 (2)	155
N1—H1C···Cl1^{ii}	0.91	2.36	3.146 (2)	145

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) *x*+1, *y*, *z*.

Fig. 1

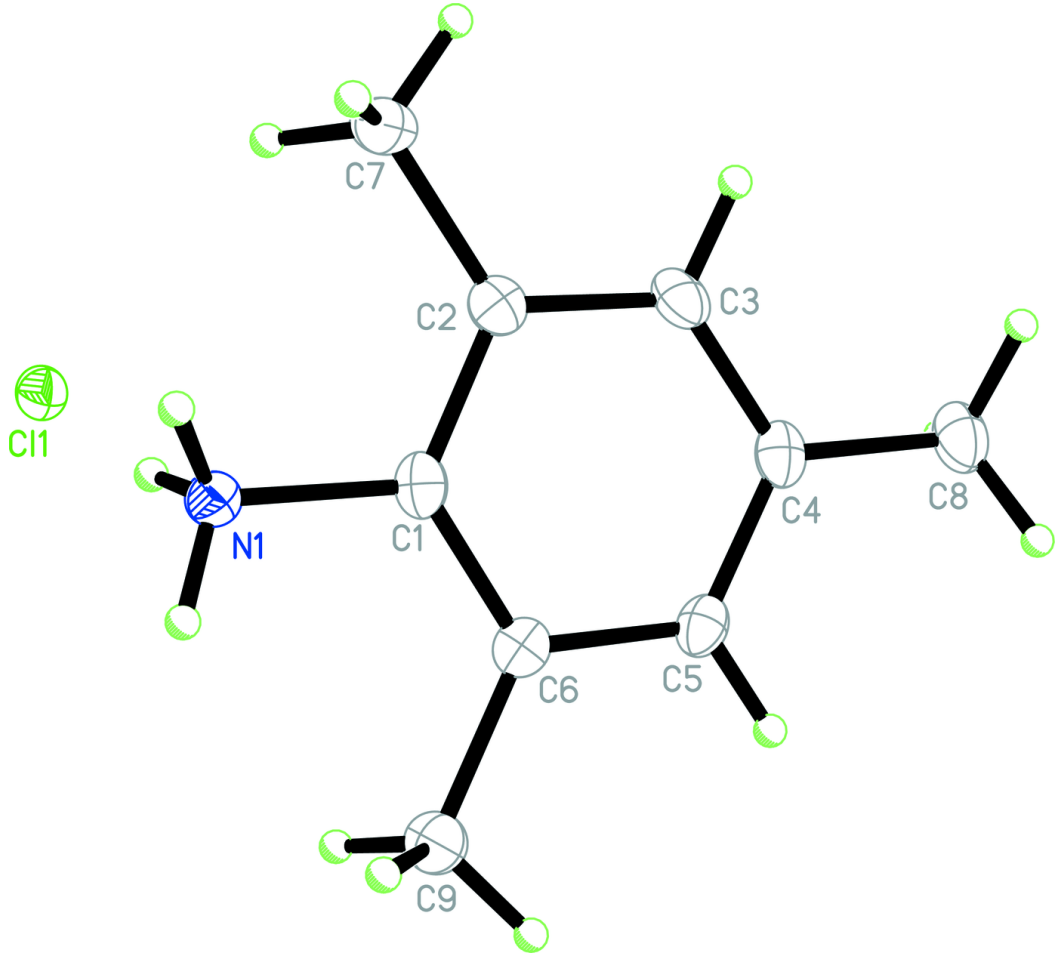

Fig. 2