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Pyridoxinium trichloroacetate

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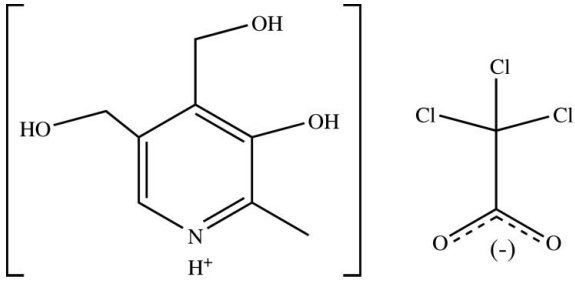
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.133; data-to-parameter ratio = 13.2.

In the title compound, $C_8H_{12}NO_3^+\cdot C_2Cl_3O_2^-$, the –$CH_2OH$ groups are twisted out of the plane of the pyridine ring. There is an intramolecular hydrogen bond between the phenol OH and the adjacent –CH_2OH group with an $S(6)$ motif. Closed $R_2^4(16)$ rings are observed around the inversion centres of the unit cell as a result of N—H···O and O—H···O interactions. Two zigzag chain $C_2^2(11)$ motifs are also formed by the hydrogen-bonding interactions of the cations and the anions.

Related literature

For related literature on hydrogen-bond motifs see Etter *et al.* (1990) and for values of bond lengths and angles see Allen *et al.* (1987). For related structures see Longo *et al.* (1982), Bacon & Plant (1980), Longo & Richardson, (1980), Franklin & Richardson (1980), Dey *et al.* (2003), Culbertson *et al.* (2003), Bonfada *et al.* (2005) and Anitha *et al.* (2006). For other related literature, see: Leklem (1990); Sabirov *et al.* (1993); Trakatellis *et al.* (1992).



Experimental

Crystal data

$C_8H_{12}NO_3^+\cdot C_2Cl_3O_2^-$
M_r = 332.56
Monoclinic, $P2_1/c$
a = 5.8552 (5) Å
b = 17.1467 (15) Å
c = 14.0988 (10) Å
β = 97.948 (17)°
V = 1401.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 293 (2) K
0.21 × 0.19 × 0.17 mm

Data collection

Nonius MACH3 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.881, T_{max} = 0.899
2906 measured reflections
2459 independent reflections
1531 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.133
S = 1.01
2459 reflections
186 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected torsion angles (°).

C3—C4—C41—O42	−21.0 (8)	C6—C5—C51—O52	0.3 (5)
C3—C4—C41—O41	10.0 (7)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O11	0.86	1.91	2.752 (4)	166
O3—H3···O41	0.82	1.83	2.544 (13)	145
O3—H3···O42	0.82	1.81	2.518 (17)	143
O41—H41···O12^{i}	0.82	1.77	2.577 (13)	166
O42—H42···O12^{i}	0.82	1.88	2.683 (14)	164
O52—H52···O11^{ii}	0.82	1.95	2.729 (4)	158

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *ORTEP-3* (Farrugia, 1997), *Mercury* (Macrae *et al.*, 2006) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2360).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Anitha, K., Athimoolam, S. & Natarajan, S. (2006). *Acta Cryst.* C**62**, o426–o428.
Bacon, G. E. & Plant, J. S. (1980). *Acta Cryst.* B**36**, 1130–1136.
Bonfada, E., de Oliveira, G. M., Back, D. F. & Lang, E. S. (2005). *Z. Anorg. Allg. Chem.* **631**, 878–881.
Bruker (2000). *SHELXTL/PC*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Culbertson, S. M., Enright, G. D. & Ingold, K. U. (2003). *Org. Lett.* **5**, 2659–2662.

  doi:10.1107/S1600536807022611

Dey, S., Banerjee, P., Gangopadhyay, S. & Vojtisek, P. (2003). *Transition Met. Chem.* **28**, 765–771.
Enraf–Nonius (1994). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Franklin, K. J. & Richardson, M. F. (1980). *Inorg. Chem.* **19**, 2107–2113.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Leklem, J. E. (1990). *Handbook of Vitamins*, edited by L. J. Machlin, 2nd ed. New York: Marcel Dekker.
Longo, J., Franklin, K. J. & Richardson, M. F. (1982). *Acta Cryst.* B**38**, 2721–2724.
Longo, J. & Richardson, M. F. (1980). *Acta Cryst.* B**36**, 2456–2458.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sabirov, V. K., Porai-Koshitz, M. A. & Struchkov, Yu. T. (1993). *Acta Cryst.* C**49**, 1611–1614.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Trakatellis, A., Dimitriadou, A., Exindari, M., Scountzou, J., Koliakos, G., Christodoulou, D., Malissiovas, N., Antoniadis, A. & Polyzoni, T. (1992). *Postgrad. Med. J.* **68**, S70–77.

supplementary materials

Acta Cryst. (2007). E**63**, o2916-o2917 [**doi:10.1107/S1600536807022611**]

Pyridoxinium trichloroacetate

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Comment

Vitamin B6, a water-soluble vitamin, is also known as pyridoxine. It is essential for both mental and physical health. Other forms of vitamin B6 include pyridoxal and pyridoxamine. Pyridoxine is involved in the production of antibodies, which protect humans against bacterial diseases. Furthermore, the combination of pyridoxine with immunosuppressive drugs improves the efficiency of that therapy (Trakatellis *et al.*, 1992). Pyridoxal phosphate can bind to steroid hormone receptors and may have a role in regulating steroid hormone action. Pyridoxal phosphate can be converted to pyridoxamine phosphate which can also serve as an enzyme cofactor (Leklem, 1990). Pyridoxine has been found to play an essential role in the nervous system and aids in the metabolism of fats, carbohydrates and proteins. The crystal structures of pyridoxine (Longo *et al.*, 1982), pyridoxinium chloride (Bacon & Plant, 1980), pyridoxamine monohydrochloride (Longo & Richardson, 1980), copper complexes of neutral pyridoxamine (Franklin & Richardson, 1980), *cis*-(oxalato-*O*,*O*')-bis (pyridoxine-N)-palladium (II) (Dey *et al.*, 2003), 6-dimethyl aminopyridoxine-α^4-(t-butyldimethylsilyl ether) (Culbertson *et al.*, 2003) and aqua-bis (2-methyl-4,5-bis(hydroxymethyl) pyridinium-3-oxalato-O,*O*')-dioxo-uranium dichloride, (Bonfada *et al.*, 2005) are already known. The crystal structure of pyridoxinium picrate was already investigated from our laboratory (Anitha *et al.*, 2006). In the present work, the crystal structure of pyridoxinie trichloroacetate is reported.

The asymmetric part of the unit cell of (I), contains a pyridoxinium cation and a trichloroacetate anion (Fig. 1). One of the $-CH_2OH$ groups, is disordered over two positions. Generally, many of the vitamin B6 structures so far determined exist as zwitterions in which the phenolic group is deprotonated and pyridine N atom is protonated (Cambridge Structural Database; Version 5.28; Allen, 2002), a form found in metal–pyridoxine complexes such as bis(μ_2–pyridoxinato)diaquatetrachlorodiiron(III) (Sabirov *et al.*, 1993). In the present structure, both the phenolic group and the pyridine N atom are protonated like pyridoxinium picrate (Anitha *et al.*, 2006) as evidenced by the C3—O3 and C—N1 bond distances (Table 1). Twisting of the $-CH_2OH$ groups is a characteristic feature of all pyridoxine complexes. Twisting in the present structure can be notified from the torsional angles involved in the $-CH_2OH$ groups (Table 1). The deviations of atoms O41, O42 and O52 from the plane of the ring are −0.166 (11), 0.537 (15) and −0.029 (6) Å, respectively.

An ntramolecular hydrogen bond forms between the phenolic OH and the adjacent $-CH_2OH$ group, generating an S(6) hydrogen-bonded graph-set motif (Etter *et al.*, 1990). This S(6) intramolecular motif is observed in many pyridoxine complexes, and is an another characterestic feature. The pyridoxinium cation is linked to the anion and forms a closed ring structure through N—H—O and O—H···O hydrogen bonds around the inversion centres of the unit cell leading to the graph-set motif of $R_2^4(16)$ (Fig 2). The disordered $-CH_2OH$ group and the phenolic –OH group are making interaction with the anion through O—H···O hydrogen bonds leading to zigzag chain $C_2^2(11)$ motif. Another $C_2^2(11)$ motif propogating along the *b* axis is seen through N—H···O and O—H···O hydrogen bonds (Table 2).

Experimental

The title compound (I), was crystallized from an aqueous mixture of pyridoxine and trichloroacetic acid in the stoichiometric ratio of 1:1 at room temperature by the technique of slow evaporation.

Refinement

All the hydrogen atoms were placed in geometrically calculated positions and included in the refinement as riding-model approximation, with O—H = 0.82 Å, C—H = 0.93–0.97 Å and N—H = 0.86 Å and U_{iso} equal to 1.2–1.5 U_{eq} of the carrier atom. In the cation, O atom in one of the $–CH_2OH$ groups is disordered over two postions with the site occupancies of 0.57 and 0.43.

Figures

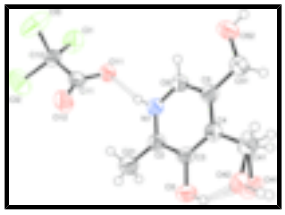

Fig. 1. The molecular structure of title compound with atom numbering scheme and 50% probability displacement ellipsoids. H-bonds are shown as dashed lines.

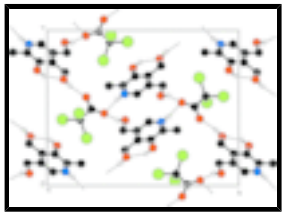

Fig. 2. A packing diagram of (I), viewed down the *a* axis. Only the major components of the disordered atoms are shown for claritly. H atoms not involved in the H-bonds (dashed lines) are removed for clarity.

Pyridoxinium trichloroacetate

Crystal data

$C_8H_{12}NO_3^+·C_2Cl_3O_2^-$	F_{000} = 680
	D_x = 1.576 Mg m^{-3}
M_r = 332.56	D_m = 1.56 (1) Mg m^{-3} D_m measured by flotation using a liquid-mixture of xylene and carbon tetrachloride
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 5.8552 (5) Å	θ = 9.6–13.6º
b = 17.1467 (15) Å	μ = 0.67 mm^{-1}
c = 14.0988 (10) Å	T = 293 (2) K
β = 97.948 (17)º	Block, colourless
V = 1401.9 (2) Å^3	0.21 × 0.19 × 0.17 mm
Z = 4	

Data collection

Nonius MACH3 diffractometer	R_{int} = 0.016
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 2.4º
T = 293(2) K	h = 0→6
ω–2θ scans	k = −1→20
Absorption correction: ψ scan (North *et al.*, 1968)	l = −16→16
T_{min} = 0.881, T_{max} = 0.899	3 standard reflections
2906 measured reflections	every 60 min
2459 independent reflections	intensity decay: none
1531 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.133	$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 1.5035P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
2459 reflections	$\Delta\rho_{max}$ = 0.45 e Å^{-3}
186 parameters	$\Delta\rho_{min}$ = −0.36 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}	Occ. (<1)
N1	0.0773 (5)	0.09533 (16)	0.09102 (19)	0.0420 (7)	
H1	0.1052	0.1338	0.0553	0.050*	
C2	−0.0916 (6)	0.1034 (2)	0.1446 (2)	0.0400 (8)	

C21	−0.2235 (7)	0.1776 (2)	0.1420 (3)	0.0578 (10)	
H21A	−0.1525	0.2157	0.1057	0.087*	
H21B	−0.2242	0.1963	0.2062	0.087*	
H21C	−0.3792	0.1686	0.1126	0.087*	
C3	−0.1321 (6)	0.0409 (2)	0.2035 (2)	0.0401 (8)	
O3	−0.3063 (5)	0.05226 (16)	0.2563 (2)	0.0581 (7)	
H3	−0.3096	0.0160	0.2940	0.087*	
C4	0.0004 (6)	−0.02650 (19)	0.2062 (2)	0.0396 (8)	
C41	−0.0325 (7)	−0.0941 (2)	0.2707 (3)	0.0554 (10)	
H41A	−0.0410	−0.1419	0.2336	0.067*	0.53
H41B	0.1005	−0.0979	0.3199	0.067*	0.53
H42A	−0.1220	−0.1348	0.2351	0.067*	0.47
H42B	0.1163	−0.1156	0.2967	0.067*	0.47
O41	−0.2380 (18)	−0.0869 (7)	0.3159 (9)	0.074 (3)	0.57
H41	−0.2351	−0.1194	0.3588	0.112*	0.57
O42	−0.146 (3)	−0.0673 (8)	0.3456 (11)	0.072 (4)	0.43
H42	−0.1816	−0.1046	0.3769	0.108*	0.43
C5	0.1720 (6)	−0.0312 (2)	0.1463 (2)	0.0406 (8)	
C51	0.3171 (7)	−0.1035 (2)	0.1441 (3)	0.0528 (10)	
H51A	0.3993	−0.1137	0.2074	0.063*	
H51B	0.2192	−0.1480	0.1249	0.063*	
O52	0.4755 (6)	−0.09238 (17)	0.0786 (2)	0.0723 (9)	
H52	0.5598	−0.1306	0.0792	0.108*	
C6	0.2066 (6)	0.0309 (2)	0.0894 (2)	0.0422 (8)	
H6	0.3201	0.0285	0.0494	0.051*	
O11	0.2154 (5)	0.20163 (14)	−0.03583 (18)	0.0530 (7)	
O12	0.2032 (6)	0.29565 (17)	0.0689 (2)	0.0767 (10)	
C11	0.2437 (6)	0.2694 (2)	−0.0072 (3)	0.0436 (8)	
C12	0.3523 (6)	0.3270 (2)	−0.0752 (3)	0.0463 (9)	
Cl1	0.3153 (2)	0.29344 (7)	−0.19483 (8)	0.0776 (4)	
Cl2	0.2266 (3)	0.41993 (7)	−0.07418 (10)	0.0905 (5)	
Cl3	0.6480 (2)	0.33330 (10)	−0.03478 (11)	0.0981 (5)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0488 (18)	0.0368 (16)	0.0421 (16)	−0.0028 (14)	0.0122 (14)	0.0037 (13)
C2	0.043 (2)	0.0382 (18)	0.0396 (18)	−0.0004 (16)	0.0068 (16)	0.0005 (15)
C21	0.059 (3)	0.048 (2)	0.070 (3)	0.008 (2)	0.020 (2)	0.009 (2)
C3	0.040 (2)	0.044 (2)	0.0379 (18)	−0.0040 (16)	0.0118 (15)	−0.0043 (15)
O3	0.0587 (17)	0.0566 (17)	0.0657 (18)	0.0067 (14)	0.0325 (14)	0.0079 (14)
C4	0.046 (2)	0.0372 (18)	0.0354 (17)	−0.0019 (16)	0.0056 (15)	−0.0001 (14)
C41	0.064 (3)	0.050 (2)	0.057 (2)	0.003 (2)	0.024 (2)	0.0113 (19)
O41	0.071 (6)	0.070 (7)	0.090 (8)	0.004 (4)	0.040 (5)	0.036 (5)
O42	0.108 (12)	0.043 (6)	0.079 (9)	0.001 (6)	0.061 (8)	0.020 (5)
C5	0.043 (2)	0.0397 (19)	0.0397 (18)	−0.0031 (16)	0.0063 (15)	−0.0029 (15)
C51	0.056 (2)	0.043 (2)	0.062 (2)	0.0059 (18)	0.019 (2)	0.0036 (18)
O52	0.081 (2)	0.0550 (18)	0.091 (2)	0.0190 (15)	0.0490 (19)	0.0064 (16)

C6	0.044 (2)	0.0398 (19)	0.046 (2)	−0.0001 (16)	0.0148 (16)	−0.0023 (16)
O11	0.0693 (18)	0.0352 (14)	0.0612 (16)	−0.0001 (12)	0.0321 (14)	0.0019 (12)
O12	0.118 (3)	0.0550 (18)	0.0680 (19)	−0.0143 (17)	0.0530 (19)	−0.0151 (15)
C11	0.045 (2)	0.041 (2)	0.047 (2)	0.0042 (17)	0.0139 (17)	0.0031 (16)
C12	0.045 (2)	0.042 (2)	0.053 (2)	0.0003 (17)	0.0116 (17)	0.0090 (17)
Cl1	0.1067 (10)	0.0787 (8)	0.0521 (6)	−0.0178 (7)	0.0271 (6)	0.0053 (6)
Cl2	0.1338 (12)	0.0476 (6)	0.0919 (9)	0.0246 (7)	0.0218 (8)	0.0187 (6)
Cl3	0.0517 (7)	0.1302 (12)	0.1074 (10)	−0.0239 (7)	−0.0065 (6)	0.0533 (9)

Geometric parameters (Å, °)

N1—C2	1.332 (4)	C41—H42A	0.9700
N1—C6	1.342 (4)	C41—H42B	0.9700
N1—H1	0.8600	O41—H41	0.8200
C2—C3	1.396 (5)	O42—H42	0.8200
C2—C21	1.486 (5)	C5—C6	1.365 (5)
C21—H21A	0.9600	C5—C51	1.505 (5)
C21—H21B	0.9600	C51—O52	1.410 (4)
C21—H21C	0.9600	C51—H51A	0.9700
C3—O3	1.357 (4)	C51—H51B	0.9700
C3—C4	1.390 (5)	O52—H52	0.8200
O3—H3	0.8200	C6—H6	0.9300
C4—C5	1.402 (5)	O11—C11	1.235 (4)
C4—C41	1.502 (5)	O12—C11	1.217 (4)
C41—O42	1.400 (15)	C11—C12	1.570 (5)
C41—O41	1.443 (12)	C12—Cl3	1.750 (4)
C41—H41A	0.9700	C12—Cl2	1.757 (4)
C41—H41B	0.9700	C12—Cl1	1.766 (4)
C2—N1—C6	124.1 (3)	H41A—C41—H42A	29.4
C2—N1—H1	118.0	H41B—C41—H42A	130.2
C6—N1—H1	118.0	O42—C41—H42B	109.6
N1—C2—C3	117.3 (3)	O41—C41—H42B	128.8
N1—C2—C21	119.9 (3)	C4—C41—H42B	109.8
C3—C2—C21	122.8 (3)	H41A—C41—H42B	81.9
C2—C21—H21A	109.5	H41B—C41—H42B	27.7
C2—C21—H21B	109.5	H42A—C41—H42B	108.4
H21A—C21—H21B	109.5	C41—O41—H41	109.5
C2—C21—H21C	109.5	C41—O42—H42	109.5
H21A—C21—H21C	109.5	C6—C5—C4	119.1 (3)
H21B—C21—H21C	109.5	C6—C5—C51	120.0 (3)
O3—C3—C4	124.3 (3)	C4—C5—C51	120.9 (3)
O3—C3—C2	114.7 (3)	O52—C51—C5	109.0 (3)
C4—C3—C2	121.0 (3)	O52—C51—H51A	109.9
C3—O3—H3	109.5	C5—C51—H51A	109.9
C3—C4—C5	118.4 (3)	O52—C51—H51B	109.9
C3—C4—C41	122.7 (3)	C5—C51—H51B	109.9
C5—C4—C41	118.9 (3)	H51A—C51—H51B	108.3
O42—C41—O41	29.2 (6)	C51—O52—H52	109.5
O42—C41—C4	108.5 (7)	N1—C6—C5	120.2 (3)

O41—C41—C4	112.7 (6)	N1—C6—H6	119.9
O42—C41—H41A	133.6	C5—C6—H6	119.9
O41—C41—H41A	109.0	O12—C11—O11	127.1 (3)
C4—C41—H41A	109.0	O12—C11—C12	116.8 (3)
O42—C41—H41B	84.3	O11—C11—C12	116.0 (3)
O41—C41—H41B	109.0	C11—C12—Cl3	107.8 (2)
C4—C41—H41B	109.0	C11—C12—Cl2	111.1 (2)
H41A—C41—H41B	107.8	Cl3—C12—Cl2	109.7 (2)
O42—C41—H42A	110.3	C11—C12—Cl1	111.9 (3)
O41—C41—H42A	82.3	Cl3—C12—Cl1	108.4 (2)
C4—C41—H42A	110.3	Cl2—C12—Cl1	107.9 (2)
C6—N1—C2—C3	1.1 (5)	C41—C4—C5—C6	−178.3 (3)
C6—N1—C2—C21	179.4 (3)	C3—C4—C5—C51	−178.4 (3)
N1—C2—C3—O3	−179.9 (3)	C41—C4—C5—C51	1.7 (5)
C21—C2—C3—O3	1.8 (5)	C6—C5—C51—O52	0.3 (5)
N1—C2—C3—C4	0.5 (5)	C4—C5—C51—O52	−179.6 (3)
C21—C2—C3—C4	−177.8 (3)	C2—N1—C6—C5	−1.3 (5)
O3—C3—C4—C5	178.6 (3)	C4—C5—C6—N1	−0.1 (5)
C2—C3—C4—C5	−1.9 (5)	C51—C5—C6—N1	179.9 (3)
O3—C3—C4—C41	−1.5 (6)	O12—C11—C12—Cl3	80.3 (4)
C2—C3—C4—C41	178.1 (3)	O11—C11—C12—Cl3	−97.6 (3)
C3—C4—C41—O42	−21.0 (8)	O12—C11—C12—Cl2	−40.0 (4)
C5—C4—C41—O42	158.9 (7)	O11—C11—C12—Cl2	142.2 (3)
C3—C4—C41—O41	10.0 (7)	O12—C11—C12—Cl1	−160.6 (3)
C5—C4—C41—O41	−170.1 (5)	O11—C11—C12—Cl1	21.5 (4)
C3—C4—C5—C6	1.6 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O11	0.86	1.91	2.752 (4)	166
O3—H3···O41	0.82	1.83	2.544 (13)	145
O3—H3···O42	0.82	1.81	2.518 (17)	143
O41—H41···O12^{i}	0.82	1.77	2.577 (13)	166
O42—H42···O12^{i}	0.82	1.88	2.683 (14)	164
O52—H52···O11^{ii}	0.82	1.95	2.729 (4)	158

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) −*x*+1, −*y*, −*z*.

Fig. 1

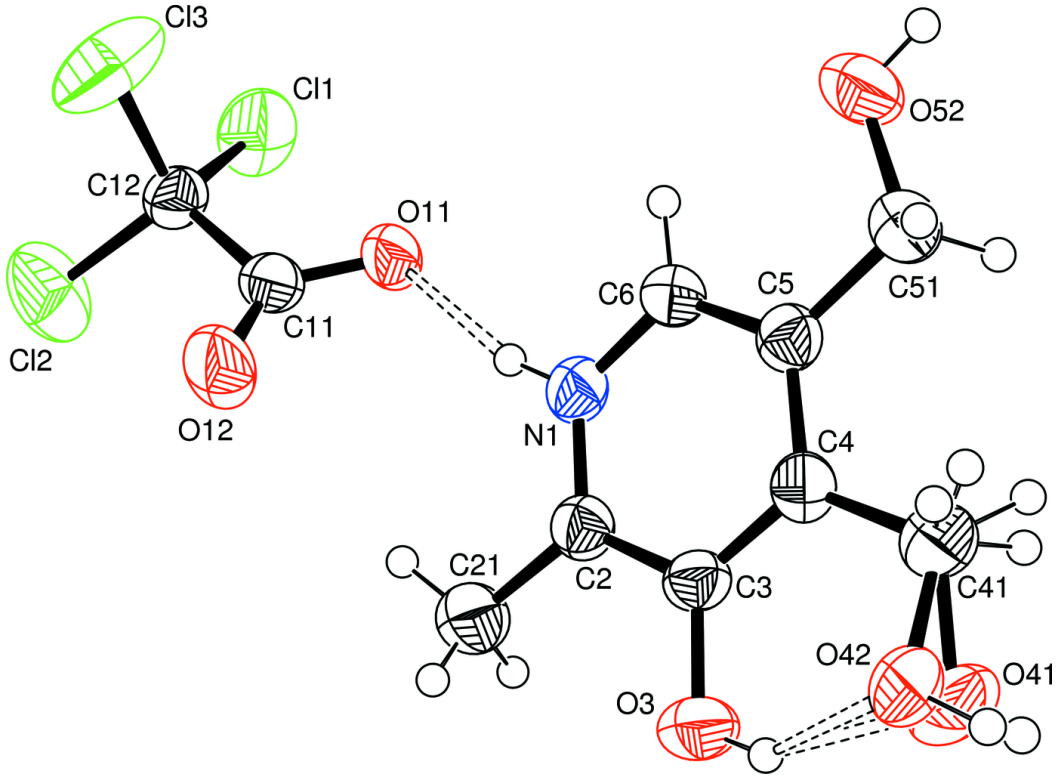
Cl3
Cl1
C12
O11
Cl2
C11
O12
C6
C5
O52
C51
N1
C4
C21
C2
C3
C41
O42
O41
O3

Fig. 2