Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Retraction of articles

This article reports the retraction of five articles published in *Acta Crystallographica Section E* between 2004 and 2011.

After further thorough investigation (see Harrison *et al.*, 2010), five articles are retracted as a result of problems with the data sets or incorrect atom assignments. Full details of all the articles are given in Table 1.

Table 1
Details of articles to be retracted, in order of publication.

Title	Reference	DOI	Refcode
Ammonium 2,6-dicarboxy-4-nitrophenolate	Sun & Nie (2004)	10.1107/S1600536804022135	PAHDUY
Triaqua(1,10-phenanthroline)sulfatocopper(II) monohydrate	An *et al.* (2007)	10.1107/S1600536807000591	HEWQUW
Diaqua-1κO,3κO-di-μ-cyanido-1:2κ^{2}N:C;2:3κ^{2}C:N-dicyanido-2κ^{2}C-bis{4,4′-dibromo-2,2′-[propane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-1κ^{4}O,N,N′,O′;3κ^{4}O,N,N′,O′-1,3-diiron(III)-2-nickel(II)	Zhang *et al.* (2008)	10.1107/S1600536808017893	SOGBOG
Bis(6-methoxy-2-{[tris(hydroxymethyl)methyl]iminomethyl}phenolato)copper(II) dihydrate	Zhang *et al.* (2009)	10.1107/S1600536808043948	ROLPAK
Oxonium picrate	Jin *et al.* (2011)	10.1107/S1600536811022574	EVILAX

References

An, Z., Wu, Y.-L., Lin, F. & Zhu, L. (2007). *Acta Cryst.* E**63**, m477–m478.
Harrison, W. T. A., Simpson, J. & Weil, M. (2010). *Acta Cryst.* E**66**, e1–e2.
Jin, S.-W., Chen, B.-X., Ge, Y.-S., Yin, H.-B. & Fang, Y.-P. (2011). *Acta Cryst.* E**67**, o1694.
Sun, Y.-X. & Nie, Y. (2004). *Acta Cryst.* E**60**, o1742–o1744.
Zhang, X., Wei, P., Dou, J., Li, B. & Hu, B. (2009). *Acta Cryst.* E**65**, m151–m152.
Zhang, X., Wei, P. & Li, B. (2008). *Acta Cryst.* E**64**, m926.

  doi:10.1107/S1600536811030649

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Oxonium picrate

Shou-Wen Jin,* Bing-Xia Chen, Yu-Shuang Ge, Hua-Bing Yin and Yu-Ping Fang

Tianmu College, ZheJiang A & F University, Lin'An 311300, People's Republic of China
Correspondence e-mail: jingaoyf@yahoo.cn

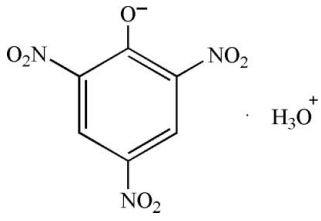
Received 18 May 2011; accepted 10 June 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.140; data-to-parameter ratio = 9.5.

The title compound, $H_3O^+\cdot C_6H_2N_3O_7^-$, consists of one picrate anion and one oxonium cation. The oxonium cation is located on a crystallographic twofold axis and both its H atoms are disordered, each over two symmetry-equivalent positions with occupancy ratios of 0.75. The picrate anions are also located on twofold axes bisecting the phenolate and *p*-nitro groups. π–π interactions between the rings of the picrates [centroid-to-centroid distances of 3.324 (2) Å] connect the anions to form stacks along the *a*-axis direction. The stacks are further joined together by the protonated water molecules through hydrogen bonds to form two-dimensional sheets extending parallel to the *ab* plane. The sheets are stacked on top of each other along the *c*-axis direction and connected through C—H···O interactions between the CH groups of the benzene rings and the picrate nitro groups, with C···O distances of 3.450 (2) Å.

Related literature

For general background to organic salts of picric acid, see Jin *et al.* (2010); Harrison *et al.* (2007); Muthamizhchelvan *et al.* (2005); Smith *et al.* (2004).



Experimental

Crystal data

$H_3O^+\cdot C_6H_2N_3O_7^-$
M_r = 247.13
Orthorhombic, *Ibca*
a = 7.1510 (6) Å
b = 19.80820 (18) Å
c = 13.50610 (12) Å
V = 1913.12 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 298 K
0.45 × 0.34 × 0.31 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.936, T_{max} = 0.951
3842 measured reflections
848 independent reflections
654 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.140
S = 1.12
848 reflections
89 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5*B*···O2^{i}	0.91 (2)	2.17 (2)	3.061 (3)	166 (5)
O5—H5*A*···O1	0.92 (2)	1.93 (2)	2.848 (2)	172 (5)
C3—H3···O3^{ii}	0.93	2.52	3.450 (2)	175

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge financial support from the Education Office Foundation of Zhejiang Province (project No. Y201017321) and from the Innovation Project of Zhejiang A & F University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2375).

References

Bruker (2002). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Harrison, W. T. A., Swamy, M. T., Nagaraja, P., Yathirajan, H. S. & Narayana, B. (2007). *Acta Cryst.* E**63**, o3892.
Jin, S. W., Zhang, W. B., Liu, L., Gao, H. F., Wang, D. Q., Chen, R. P. & Xu, X. L. (2010). *J. Mol. Struct.* **975**, 128–136.
Muthamizhchelvan, C., Saminathan, K., Fraanje, J., Peschar, R. & Sivakamar, K. (2005). *Anal. Sci.* **21**, x61–x62.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, G., Wermuth, U. D. & Healy, P. C. (2004). *Acta Cryst.* E**60**, o1800–o1803.

supporting information

Acta Cryst. (2011). E**67**, o1694 [doi:10.1107/S1600536811022574]

Oxonium picrate

Shou-Wen Jin, Bing-Xia Chen, Yu-Shuang Ge, Hua-Bing Yin and Yu-Ping Fang

S1. Comment

It is well known that picric acid is used primarily to prepare explosives, and as an intermediate to manufacture dyes. As a strong organic acid, picric acid forms salts with many N-containing organic bases (Smith *et al.*, 2004; Harrison *et al.*, 2007; Muthamizhchelvan *et al.*, 2005). As an extension of our study concerning organic salts based on picric acid (Jin *et al.*, 2010), we herein report the crystal structure of oxonium picrate.

The single crystal of the title compound (Fig. 1) with the formula $C_6H_5N_3O_8$ was obtained by recrystallization of picric acid and 2-chloropyridine from a methanol solution. However the 2-chloropyridine molecules do not appear in the title compound. X-ray diffraction analysis indicated that in the title compound there are one protonated water molecule, and one picrate. The OH group of the picric acid is ionized and the proton is transferred to the water molecule. In the title compound all of the bond distances and angles are in the normal range. The oxonium cation is located on a crystallographic two-fold axis and both its H atoms are disordered over each two symmetry equivalent positions with occupancy rates of 0.75 each. The benzene ring of the picrate is almost planar. The *ortho*-nitro groups (N1—O2—O3, and N1A—O2A—O3A) deviate from the benzene ring plane and have a dihedral angle of 25.6 (2)° with the benzene plane, whereas the *para*-nitro group lies almost in the benzene plane [with a dihedral angle of 2.0 (1)° between the N2—O4—O4A group and the benzene ring]. These structural data are similar to those in other structurally described picrates (Muthamizhchelvan *et al.*, 2005).

π–*π* Interactions between the phenyl rings of the picrates (with Cg–Cg distances of 3.324 (2) Å) connect the picrate anions to form stacks along the *a* axis direction. Within one stack molecules alternate and are arranged in an antiparallel fashion. The one-dimensional picrate stacks are further linked together by the oxonium ions to form a two-dimensional sheet structure when it is viewed from the *c* axis direction (Fig. 2). The sheets are further stacked along the *c* axis direction through CH···O interactions between CH of the benzene rings and the nitro groups of the picrates with C···O distances of 3.450 (2) Å to form a three-dimensional structure.

S2. Experimental

Crystals of oxonium picrate were formed by slow evaporation of its methanol solution at room temperature. Picric acid (23 mg, 0.10 mmol) was dissolved in 4 ml of methanol, and 2-chloropyridine (11 mg, 0.10 mmol) was added to the methanol solution. The solution was then filtered into a test tube and left standing at room temperature. After about one week yellow block crystals were obtained.

S3. Refinement

H atoms H5A and H5B bonded to the oxonium O atom were located in a difference Fourier map and refined isotropically. The oxonium cation is located on a crystallographic two-fold axis and both H5A and H5B are disordered over each two symmetry equivalent positions, and both have an occupancy of 0.75. Other H atoms were positioned geometrically with

C—H = 0.93 Å for aromatic H atoms, and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

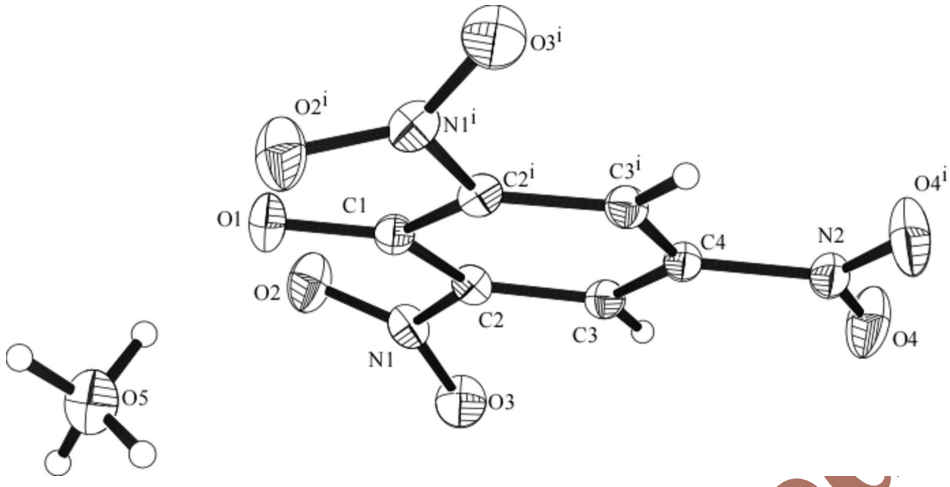


Figure 1

The structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code: (i) -*x* + 1/2, *y*, -*z*.

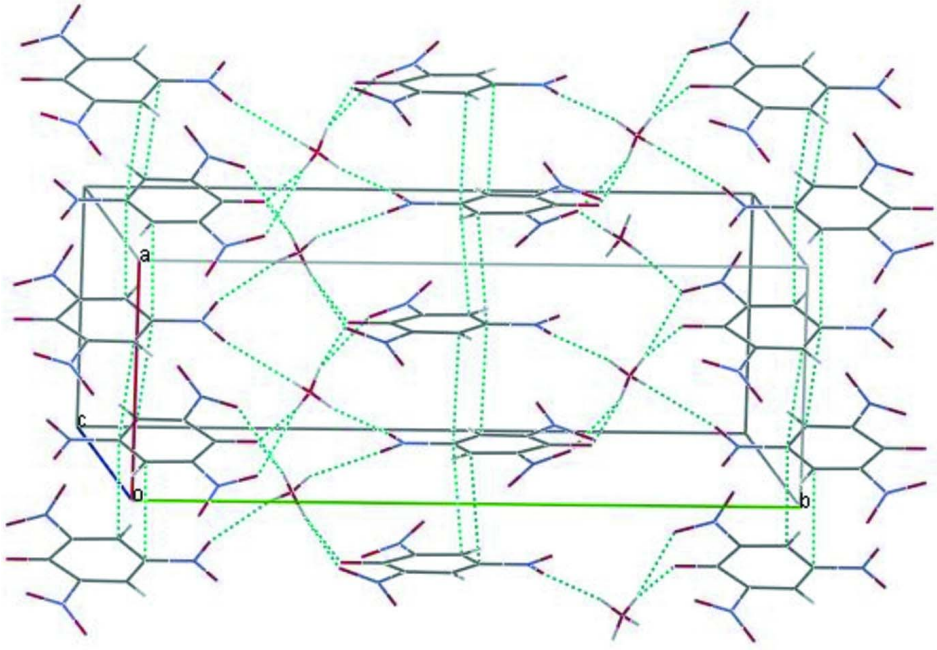


Figure 2

Two-dimensional sheet structure formed through hydrogen bonds which is viewed along the *c* axis direction. The blue dashed lines represent O—H···O and π–π interactions.

Oxonium 2,4,6-trinitrophenolate

Crystal data

$H_3O^+ \cdot C_6H_2N_3O_7^-$
M_r = 247.13
Orthorhombic, *Ibca*

a = 7.1510 (6) Å
b = 19.80820 (18) Å
c = 13.50610 (12) Å

V = 1913.12 (16) Å^3
Z = 8
F(000) = 1008
D_x = 1.716 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1867 reflections

θ = 1.5–25.0°
μ = 0.16 mm^{-1}
T = 298 K
Block, yellow
0.45 × 0.34 × 0.31 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.936, T_{max} = 0.951

3842 measured reflections
848 independent reflections
654 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 25.0°, θ_{min} = 2.1°
h = −8→8
k = −22→23
l = −16→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.140
S = 1.12
848 reflections
89 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 3.1011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.039 (4)

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 $F^2 > \sigma(F^2)$ 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	0.3838 (3)	0.11353 (11)	0.17214 (16)	0.0328 (7)	
N2	0.2500	−0.09568 (15)	0.0000	0.0361 (8)	
O1	0.2500	0.18513 (12)	0.0000	0.0369 (8)	
O2	0.3327 (3)	0.17048 (10)	0.19325 (15)	0.0502 (7)	
O3	0.4947 (3)	0.08131 (11)	0.22328 (15)	0.0497 (7)	
O4	0.3100 (4)	−0.12491 (10)	0.07276 (19)	0.0630 (8)	
O5	0.0000	0.2500	0.1338 (2)	0.0489 (9)	
H5A	0.089 (6)	0.233 (3)	0.091 (3)	0.073*	0.75
H5B	0.052 (7)	0.279 (2)	0.177 (3)	0.073*	0.75

C1	0.2500	0.12179 (17)	0.0000	0.0270 (8)
C2	0.3107 (4)	0.08102 (13)	0.08289 (17)	0.0270 (7)
C3	0.3134 (4)	0.01147 (13)	0.08305 (17)	0.0286 (7)
H3	0.3569	−0.0122	0.1378	0.034*
C4	0.2500	−0.02233 (17)	0.0000	0.0282 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0376 (14)	0.0358 (13)	0.0250 (12)	−0.0067 (10)	−0.0014 (10)	0.0001 (9)
N2	0.0350 (19)	0.0269 (16)	0.046 (2)	0.000	0.0059 (15)	0.000
O1	0.0587 (19)	0.0226 (13)	0.0295 (14)	0.000	0.0085 (13)	0.000
O2	0.0748 (17)	0.0372 (12)	0.0387 (12)	0.0047 (10)	−0.0128 (11)	−0.0126 (9)
O3	0.0569 (15)	0.0538 (13)	0.0385 (12)	0.0006 (11)	−0.0193 (10)	0.0019 (9)
O4	0.096 (2)	0.0300 (12)	0.0629 (15)	0.0117 (11)	−0.0140 (14)	0.0089 (10)
O5	0.061 (2)	0.0414 (17)	0.0443 (18)	−0.0008 (15)	0.000	0.000
C1	0.0273 (19)	0.0275 (18)	0.0260 (18)	0.000	0.0050 (14)	0.000
C2	0.0287 (14)	0.0305 (14)	0.0217 (13)	−0.0032 (10)	0.0009 (10)	−0.0014 (9)
C3	0.0279 (14)	0.0303 (14)	0.0275 (13)	0.0010 (11)	−0.0001 (11)	0.0051 (10)
C4	0.0262 (19)	0.0248 (17)	0.0337 (19)	0.000	0.0041 (15)	0.000

Geometric parameters (Å, º)

N1—O2	1.220 (3)	O5—H5B	0.91 (2)
N1—O3	1.230 (3)	C1—C2	1.447 (3)
N1—C2	1.463 (3)	C1—C2^{i}	1.447 (3)
N2—O4	1.219 (3)	C2—C3	1.378 (4)
N2—O4^{i}	1.219 (3)	C3—C4	1.383 (3)
N2—C4	1.453 (5)	C3—H3	0.9300
O1—C1	1.255 (4)	C4—C3^{i}	1.383 (3)
O5—H5A	0.92 (2)		
O2—N1—O3	122.8 (2)	C3—C2—C1	124.3 (2)
O2—N1—C2	119.5 (2)	C3—C2—N1	115.7 (2)
O3—N1—C2	117.7 (2)	C1—C2—N1	119.9 (2)
O4—N2—O4^{i}	123.3 (3)	C2—C3—C4	118.6 (2)
O4—N2—C4	118.37 (17)	C2—C3—H3	120.7
O4^{i}—N2—C4	118.37 (17)	C4—C3—H3	120.7
H5A—O5—H5B	111 (5)	C3^{i}—C4—C3	122.1 (3)
O1—C1—C2	123.92 (15)	C3^{i}—C4—N2	118.97 (16)
O1—C1—C2^{i}	123.92 (15)	C3—C4—N2	118.97 (16)
C2—C1—C2^{i}	112.2 (3)		
O1—C1—C2—C3	−179.13 (18)	C1—C2—C3—C4	−1.7 (3)
C2^{i}—C1—C2—C3	0.87 (18)	N1—C2—C3—C4	−178.59 (19)
O1—C1—C2—N1	−2.4 (3)	C2—C3—C4—C3^{i}	0.81 (17)
C2^{i}—C1—C2—N1	177.6 (3)	C2—C3—C4—N2	−179.19 (17)
O2—N1—C2—C3	−155.8 (3)	O4—N2—C4—C3^{i}	178.42 (19)

O3—N1—C2—C3	23.8 (3)	O4^{i}—N2—C4—C3^{i}	−1.58 (19)
O2—N1—C2—C1	27.2 (3)	O4—N2—C4—C3	−1.58 (19)
O3—N1—C2—C1	−153.3 (2)	O4^{i}—N2—C4—C3	178.42 (19)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*B*···O2^{ii}	0.91 (2)	2.17 (2)	3.061 (3)	166 (5)
O5—H5*A*···O1	0.92 (2)	1.93 (2)	2.848 (2)	172 (5)
C3—H3···O3^{iii}	0.93	2.52	3.450 (2)	175

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