Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Er	1/4	1/4	3/4	0.0311 (8)
N1	0.2851 (6)	0.3432 (6)	0.9631 (17)	0.036 (3)
N2	0.1808 (6)	0.4047 (6)	0.9883 (22)	0.042 (3)
C1	0.2479 (9)	0.4002 (7)	0.9824 (22)	0.039 (4)
C2	0.2890 (7)	0.4583 (7)	1.0024 (26)	0.038 (4)
C3	0.2766 (9)	0.5243 (8)	1.0124 (27)	0.053 (5)
C4	0.3322 (9)	0.5666 (9)	1.0289 (32)	0.066 (6)
C5	0.3963 (9)	0.5434 (8)	1.0333 (31)	0.063 (5)
C6	0.4077 (9)	0.4774 (8)	1.0206 (29)	0.056 (5)
C7	0.3546 (7)	0.4338 (7)	1.0054 (24)	0.040 (4)
C8	0.3502 (8)	0.3605 (8)	0.9846 (21)	0.040 (4)

Table 2. *Selected geometric parameters* (Å, °)

Er—N1	2.410 (11)	C2—C7	1.39 (2)
N1—C8	1.35 (2)	C3—C4	1.40 (2)
N1—C1	1.36 (2)	C4—C5	1.36 (2)
N2—C1	1.34 (2)	C5—C6	1.34 (2)
N2—C8^{i}	1.36 (2)	C6—C7	1.37 (2)
C1—C2	1.42 (2)	C7—C8	1.47 (2)
C2—C3	1.34 (2)		
N1^{ii}—Er—N1^{i}	79.4 (6)	C3—C2—C7	121.2 (14)
N1^{ii}—Er—N1^{iii}	71.1 (3)	C7—C2—C1	104.8 (12)
N1^{i}—Er—N1^{iii}	140.4 (6)	C2—C3—C4	116.6 (16)
N1^{ii}—Er—N1^{iv}	82.9 (6)	C5—C4—C3	122.9 (16)
N1^{iii}—Er—N1^{v}	110.7 (5)	C6—C5—C4	119.5 (17)
N1^{iv}—Er—N1^{v}	146.3 (6)	C5—C6—C7	119.7 (17)
C8—N1—C1	107.4 (13)	C6—C7—C2	120.1 (14)
C1—N2—C8^{i}	123.3 (12)	C2—C7—C8	107.0 (12)
N2—C1—N1	126.9 (14)	N1—C8—N2^{vi}	127.8 (13)
N1—C1—C2	111.9 (14)	N1—C8—C7	108.7 (14)

Symmetry codes: (i) $\frac{1}{2}-y, x, z$; (ii) $\frac{1}{2}-y, \frac{1}{2}-x, \frac{3}{2}-z$; (iii) $x, \frac{1}{2}-y, \frac{3}{2}-z$; (iv) $\frac{1}{2}-x, \frac{1}{2}-y, z$; (v) $\frac{1}{2}-x, y, \frac{3}{2}-z$; (vi) $y, \frac{1}{2}-x, z$.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1993). Cell refinement: *CAD-4/PC*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

We thank Professors P. Fulde (MPI Dresden) and Hk. Müller-Buschbaum (University of Kiel) for providing us with facilities, and U. Cornelissen for crystal measurement. This work was supported by the Fonds der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: JZ1021). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chabach, D., Lachkar, M., de Cian, A., Fischer, J. & Weiss, R. (1992). *New J. Chem.* **16**, 431–433, and references therein.
Cian, A. de, Moussavi, M., Fischer, J. & Weiss, R. (1985). *Inorg. Chem.* **24**, 3162–3167.
Darovskikh, A. N., Frank-Kamenetskaya, O. V. & Fundamenskii, V. S. (1986). *Sov. Phys. Crystallogr.* **31**, 534–537.
Darovskikh, A. N., Frank-Kamenetskaya, O. V., Fundamenskii, V. S. & Golubev, A. M. (1986). *Sov. Phys. Crystallogr.* **31**, 165–168.
Darovskikh, A. N., Tsytsenko, A. K., Frank-Kamenetskaya, O. V., Fundamenskii, V. S. & Moskalev, P. N. (1984). *Sov. Phys. Crystallogr.* **29**, 273–276.
Darovsky, A., Keserashvili, V., Harlow, R. & Mutikainen, I. (1994). *Acta Cryst.* B**50**, 582–588.
Darovsky, A., Wu, L. Y., Lee, P. & Sheu, H. S. (1991). *Acta Cryst.* C**47**, 1836–1838.
Enraf–Nonius (1993). *CAD-4/PC Software*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Guillaud, G., Al Sadoun, M., Maitrot, M., Simon, J. & Bouvet, M. (1990). *Chem. Phys. Lett.* **167**, 503–506.
Homborg, H. & Teske, Chr. L. (1985). *Z. Anorg. Allg. Chem.* **527**, 45–61.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Maitrot, M., Guillaud, G., Boudjema, B., Andre, J.-J., Strzelecka, H., Simon, J. & Even, R. (1987). *Chem. Phys. Lett.* **133**, 59–62.
Marks, T. J. (1990). *Angew. Chem.* **102**, 886–908; *Angew. Chem. Int. Ed. Engl.* **29**, 857–888.
Mossoyan-Deneux, M., Benlian, D., Pierrot, M., Fournal, A. & Sorbier, J. P. (1985). *Inorg. Chem.* **24**, 1878–1882.
Safarpour Haghighi, M., Rath, M., Rotter, H. W. & Homborg, H. (1993). *Z. Anorg. Allg. Chem.* **619**, 1887–1896.
Safarpour Haghighi, M., Teske, Chr. L. & Homborg, H. (1992). *Z. Anorg. Allg. Chem.* **608**, 73–80.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
Sokolova, T. N., Lomova, T. N., Morozov, V. V. & Berezin, B. D. (1994). *Russ. J. Coord. Chem.* **20**, 603–606.
Takahashi, K., Itoh, M., Tomita, Y., Nojima, K., Kasuga, K. & Isa, K. (1993). *Chem. Lett.* pp. 1915–1918.
Turek, T., Petit, P., Andre, J.-J., Simon, J., Even, R., Boudjema, B., Guillaud, G. & Maitrot, M. (1987). *J. Am. Chem. Soc.* **109**, 5119–5121.

Acta Cryst. (1995). C**51**, 1128–1130

Potassium Hydrogen Phthalate Hemiperhydrate

BENSON M. KARIUKI

Department of Chemistry, University of Liverpool, PO Box 147, Liverpool L69 3BX, England

WILLIAM JONES

Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge CB2 1EW, England

(*Received* 5 *May* 1994; *accepted* 2 *November* 1994)

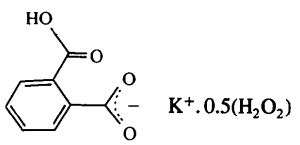
Abstract

The crystal structure of the title compound, $K^+.(HO_2C—C_6H_4—CO_2)^-.0.5H_2O_2$, is layered with eight O atoms around the cation. Anion–anion and anion–hydrogen

peroxide hydrogen bonding occurs. A short oxygen–oxygen bond distance is observed for the hydrogen peroxide.

Comment

Potassium hydrogen phthalate hemiperhydrate, (I), was first observed as an undesirable contaminant during the preparation of a solid peroxy acid salt derivative of phthalic acid. This was during a study of the reactivity of organic salts in the solid state (Kariuki, 1990). The structure was determined in order to establish its identity.



(I)

The crystal structure is composed of potassium cations, hydrogen phthalate anions and hydrogen peroxide molecules of crystallization. There are two ion pairs per hydrogen peroxide molecule. The angles between the plane through the phenyl ring and the $—CO_2$ groups are 17.82 (44)° for the carboxylic and 73.82 (23)° for the carboxylate moieties. The interplanar angle between the $—CO_2$ groups is 67.91 (38)°. Each carboxylate is hydrogen bonded to a carboxylic group of the neighbouring anion and a hydrogen peroxide molecule.

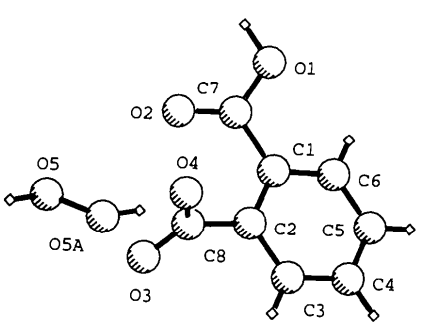


Fig. 1. View of the title compound showing the atomic numbering scheme.

The hydrogen peroxide molecule lies on a centre of symmetry. The O—O bond distance is shorter than 1.481 (9) Å, the value found in hydrogen peroxide dihydrate (Olovsson & Templeton, 1960). Short O—O bonds in hydrogen peroxide molecules have also been observed in other solids, *e.g.* in urea hydrogen peroxide (Fritchie & McMullan, 1981), dipotassium oxalate monoperhydrate (Pedersen & Kvick, 1990) and ammonium oxalate monoperhydrate (Pedersen, 1972*a*). Possible explanations for this shortening include random substitution of hydrogen peroxide by water molecules, as well as thermal motion (Pedersen, 1972*b*).

In the crystal structure, sheets composed of cations and hydrogen peroxide separate double layers of anions. The general packing features are similar to those observed in other hydrogen phthalate structures (Kariuki & Jones, 1992).

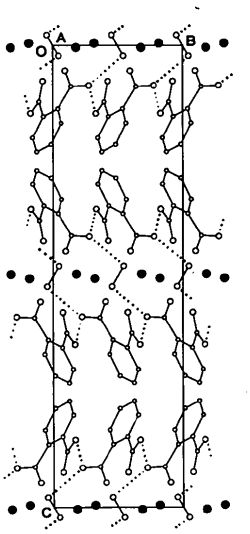


Fig. 2. The structure viewed down the *a* axis. H atoms have been omitted for clarity and the filled circles are K^+. The dotted lines represent hydrogen bonds.

Experimental

Crystals of good quality were obtained by dissolving potassium hydrogen phthalate in a 35% *w/w* solution of hydrogen peroxide in water and allowing slow evaporation at 295 K.

Crystal data

$K^+.C_8H_5O_4^-.0.5H_2O_2$	Mo $K\alpha$ radiation
M_r = 221.23	λ = 0.71069 Å
Orthorhombic	Cell parameters from 25 reflections
Pbca	θ = 6–12°
a = 9.4770 (10) Å	μ = 0.554 mm^{-1}
b = 7.4630 (10) Å	T = 293 (2) K
c = 26.727 (3) Å	Prism
V = 1890.3 (4) $Å^3$	0.3 × 0.1 × 0.1 mm
Z = 8	Colourless, transparent
D_x = 1.555 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 24.95°
$2\theta/\omega$ scans	$h = 0 \rightarrow 11$
Absorption correction: none	$k = 0 \rightarrow 8$
1658 measured reflections	$l = 0 \rightarrow 31$
1658 independent reflections	3 standard reflections monitored every 100 reflections
806 observed reflections [$I > 2\sigma(I)$]	frequency: 180 min
	intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0449$
$wR(F^2) = 0.0859$
$S = 1.262$
1654 reflections
139 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.246$ e Å^{-3}
$\Delta\rho_{min} = -0.255$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
K1	0.79525 (9)	0.18027 (11)	0.49537 (3)	0.0398 (3)
O1	0.2213 (3)	0.6690 (5)	0.64658 (9)	0.0523 (8)
O2	0.3278 (3)	0.5759 (4)	0.57715 (9)	0.0452 (8)
O3	0.6427 (3)	0.4034 (4)	0.55458 (9)	0.0458 (8)
O4	0.4818 (3)	0.2225 (3)	0.58654 (10)	0.0394 (7)
C1	0.4563 (4)	0.5718 (5)	0.65344 (12)	0.0280 (9)
C2	0.5656 (4)	0.4620 (5)	0.63671 (12)	0.0270 (9)
C3	0.6823 (4)	0.4358 (5)	0.66743 (13)	0.0383 (10)
C4	0.6884 (4)	0.5149 (6)	0.71448 (14)	0.0464 (11)
C5	0.5821 (5)	0.6246 (6)	0.73022 (14)	0.0452 (12)
C6	0.4679 (4)	0.6550 (5)	0.70019 (13)	0.0375 (10)
C7	0.3303 (4)	0.6059 (5)	0.62196 (14)	0.0337 (10)
C8	0.5624 (4)	0.3584 (5)	0.58864 (13)	0.0273 (9)
O5	0.4811 (4)	0.0450 (4)	0.47762 (11)	0.0799 (12)

Table 2. *Selected geometric parameters* (Å, °)

K1—O3^{i}	2.668 (3)	O2—C7	1.219 (4)
K1—O3	2.714 (3)	O3—C8	1.233 (4)
K1—O2^{ii}	2.740 (3)	O4—C8	1.271 (4)
K1—O5^{ii}	2.798 (3)	C1—C7	1.483 (5)
K1—O2^{iii}	2.903 (3)	C2—C8	1.500 (5)
K1—O4^{ii}	2.906 (3)	C—C(ring-mean)	1.385 (5)
K1—O5	3.180 (4)	O5—O5^{iv}	1.418 (6)
K1—O5^{iv}	3.194 (4)	O1—O4^{v}	2.538 (4)
O1—C7	1.312 (4)	O5—O4^{iv}	2.655 (4)
C2—C1—C7	121.2 (3)	O2—C7—C1	122.8 (4)
C6—C1—C7	119.6 (3)	O1—C7—C1	114.3 (3)
C3—C2—C8	116.7 (3)	O3—C8—O4	123.8 (3)
C1—C2—C8	124.2 (3)	O3—C8—C2	118.6 (3)
O2—C7—O1	122.9 (4)	O4—C8—C2	117.5 (3)

Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, z$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (iii) $1 - x, 1 - y, 1 - z$; (iv) $1 - x, -y, 1 - z$; (v) $\frac{1}{2} - x, \frac{1}{2} + y, z$.

All non-H atoms were assigned anisotropic displacement parameters. The positions of the ring H atoms were fixed geometrically and the difference-Fourier synthesis map revealed the locations of the carboxylic and hydrogen peroxide protons.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992). Molecular graphics: *DTMM* (Crabbe & Appleyard, 1991). Software used to prepare material for publication: *SHELXL*92.

Financial support by Solvay Interox PLC is appreciated.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1120). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Crabbe, M. J. C. & Appleyard, J. R. (1991). *DTMM. Desktop Molecular Modeller* 2.0. Oxford Univ. Press.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fritchie, C. J. Jr & McMullan, R. K. (1981). *Acta Cryst.* B**37**, 1086–1091.
Kariuki, B. M. (1990). PhD thesis, Univ. of Cambridge, England.
Kariuki, B. M. & Jones, W. (1992). *Mol. Cryst. Liq. Cryst.* **211**, 233–255.
Olovsson, I. & Templeton, D. H. (1960). *Acta Chem. Scand.* pp. 1325–1332.
Pedersen, B. F. (1972*a*). *Acta Cryst.* B**28**, 746–754.
Pedersen, B. F. (1972*b*). *Acta Cryst.* B**28**, 1014–1016.
Pedersen, B. F. & Kvick, A. (1990). *Acta Cryst.* C**46**, 21–23.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1992). *SHELXL*92. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.