the mirror plane and it is possible that the O atoms are very slightly disordered on either side of the mirror plane. Refinement was attempted in the non-centrosymmetric $Pna2_1$ space group, removing the requirement for mirror symmetry, but as the refinement was completely unstable even with heavy damping, this was abandoned.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *CAD-4-PC Software*. Data reduction: *DATRD*2 in *NRCVAX*96 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*96. Program(s) used to refine structure: *NRCVAX*96 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*96, *ORTEP*II (Johnson, 1976), *PLATON* (Spek, 1996*a*) and *PLUTON* (Spek 1996*b*). Software used to prepare material for publication: *NRCVAX*96, *SHELXL*93 and *WordPerfect* macro *PREPCIF* (Ferguson, 1996).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AB1456). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-(*n*-Butyl)-3,4-dibromomaleimide

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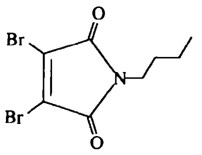
Abstract

The X-ray structure analysis of the title compound, $C_8H_9Br_2NO_2$, was carried out. In the unit cell, the molecules are stacked with their C=C double bonds at close intermolecular distances of approximately 3.6 Å. Conjugation in the imide ring is not significant.

Comment

The (2+2) photodimerization of maleic anhydride in the solid state is completely inhibited at 278 K and the crystal structure analysis of the monomer indicates that no parallel double bonds or intermolecular distances are adequate for photodimerization (Marsh, Ubell & Wilcox, 1962). In contrast, however, photodimerization of maleimide derivatives in the crystalline state takes place readily (Boens, De Schryver & Smets, 1975).

N-(*n*-Butyl)-3,4-dibromomaleimide, (I), was obtained as a by-product during the synthesis of *N*-(*n*-butyl)-3,4-dibromo-2-oxopyrrolium 5-oxide. The packing of compound (I) shows that the distance between C=C double bonds of neighbouring maleimide rings is similar to the sum of the van der Waals radii of carbon (1.7 Å). It seems likely that the packing may facilitate polymerization parallel to the *a* axis [C2···C2^{i} 3.51 (2) and C3···O1^{i} 3.66 (2) Å; symmetry code: (i) $1-x$, $2-y$, $1-z$].



(I)

The *ORTEP* (Johnson, 1965) plot of the title molecule with the numbering scheme is shown in Fig. 1. The C2—C3 distance of 1.305 (8) Å is comparable to that in maleic anhydride [1.303 Å; Marsh, Ubell & Wilcox, 1962]. The mean length of the single C—C bond in the maleimide ring [1.441 (8) Å] is comparable

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to that in 1,3-butadiene [1.455 Å; Allen *et al.*, 1987]. The C1—C2 and C3—C4 bond lengths are similar to the *n*-butyl single C—C bond lengths [1.501 (10) Å]. The bond lengths are slightly longer for C1—C2 and C3—C4 and shorter for C2—C3, N—C1 and N—C2 compared with those in *N*-(2,6-dimethylphenyl)chloromaleimide (Vrabel, Kello, Lokaj & Konecny, 1989).

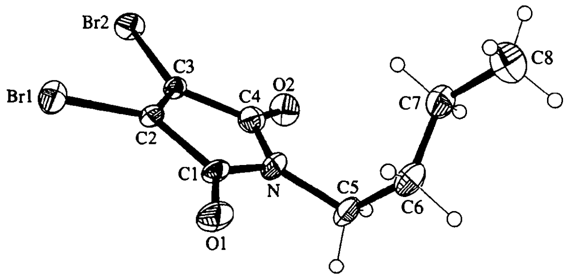

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

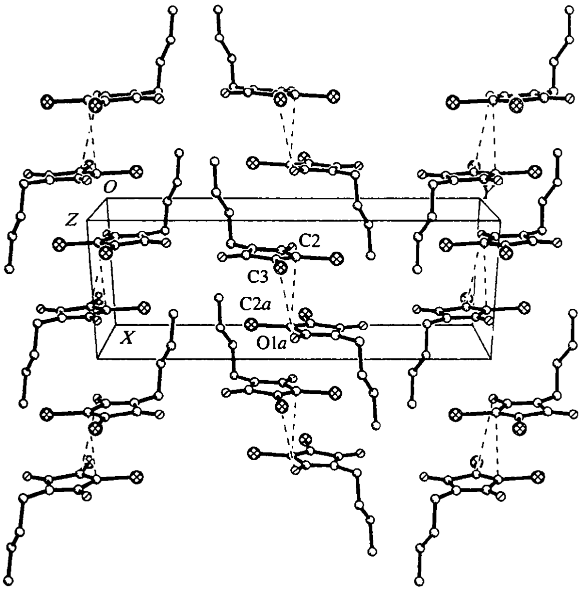

Fig. 2. Packing diagram viewed down the *c* axis.

Experimental

The title compound was synthesized by a procedure analogous to Brockmann's method (Brockmann & Tour, 1995). The product from the reaction of *N*-(*n*-butyl)pyrrole with *n*-bromosuccinimide in THF at 273 K was directly oxidized by nitric acid without separation. Recrystallization from methanol yielded yellowish and colourless crystals. A colourless crystal was used for X-ray structure analysis.

Crystal data

$C_8H_9Br_2NO_2$
$M_r = 310.98$
Monoclinic
$P2_1/c$
$a = 7.405\ (2)$ Å
$b = 18.481\ (2)$ Å
$c = 8.1246\ (9)$ Å
$\beta = 111.14\ (1)°$
$V = 1037.1\ (3)$ Å^3
$Z = 4$
$D_x = 1.992$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12–13°$
$\mu = 7.784$ mm^{-1}
$T = 300\ (2)$ K
Parallelepiped
0.57 × 0.50 × 0.43 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.018$, $T_{\max} = 0.035$
1964 measured reflections
1822 independent reflections

1353 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.0584$
$\theta_{\max} = 24.97°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 9$
3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0456$
$wR(F^2) = 0.1095$
$S = 1.092$
1822 reflections
118 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.0218P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.543$ e Å^{-3}
$\Delta\rho_{\min} = -0.766$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Br1—C2	1.848 (6)	C1—C2	1.499 (8)
Br2—C3	1.838 (6)	C2—C3	1.305 (8)
O1—C1	1.199 (7)	C3—C4	1.518 (8)
O2—C4	1.206 (8)	C5—C6	1.510 (9)
N—C4	1.386 (8)	C6—C7	1.487 (9)
N—C1	1.379 (8)	C7—C8	1.506 (11)
N—C5	1.466 (8)		
C4—N—C1	111.1 (5)	C2—C3—C4	107.7 (5)
C4—N—C5	125.2 (5)	C2—C3—Br2	130.1 (5)
C1—N—C5	123.6 (5)	C4—C3—Br2	122.2 (4)
O1—C1—N	126.5 (5)	O2—C4—N	126.8 (6)
O1—C1—C2	128.1 (6)	O2—C4—C3	127.5 (6)
N—C1—C2	105.4 (5)	N—C4—C3	105.7 (5)
C3—C2—C1	110.0 (5)	N—C5—C6	112.7 (5)
C3—C2—Br1	129.5 (5)	C7—C6—C5	113.2 (6)
C1—C2—Br1	120.5 (4)	C6—C7—C8	113.7 (6)

Data collection: *CAD*-4 *Manual* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *Manual*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1987) and *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: TA1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hydronium 2-Carboxybenzenesulfonate

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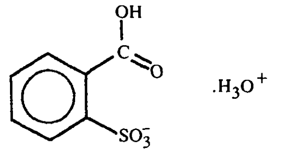
(*Received 24 June 1996; accepted 11 December 1996*)

Abstract

2-Sulfobenzoic acid hydrate exists as the title salt, oxonium 2-carboxybenzenesulfonate, $H_3O^+.C_7H_5O_5S^-$, and is isomorphous with the ammonium derivative.

Comment

2-Sulfobenzoic acid as procured from commercial sources (Aldrich Chemical Company) has the formula $HO_3SC_6H_4CO_2H.xH_2O$, where x is probably equal to 3, as implied from the trihydrated product obtained by crystallizing ammonium hydrogen 2-sulfobenzoate (Okaya, 1967) from an aqueous acidic medium (Attig & Mootz, 1976). The commercial acid, when recrystallized from an ethanol solution containing excess dicyclohexylamine, afforded dicyclohexylammonium hydrogen 2-sulfobenzoate dihydrate (Ng, 1995). In the present study, the acid was recrystallized from ethanol to give 2-sulfobenzoic acid monohydrate, which formally exists as hydronium 2-carboxybenzenesulfonate, (I).



(I)

The hydrogen 2-sulfobenzoate anions are linked into a chain along the b axis by a strong hydrogen bond involving the carboxyl O5 and sulfonyl O3 atoms [O5···O3 2.661 (3) Å]. The H_3O^+ cations surround the chains and are hydrogen bonded to adjacent chains [O···O 2.890 (3)–3.060 (3) Å] resulting in a network structure. The title compound is isomorphous with the ammonium hydrogen analog.

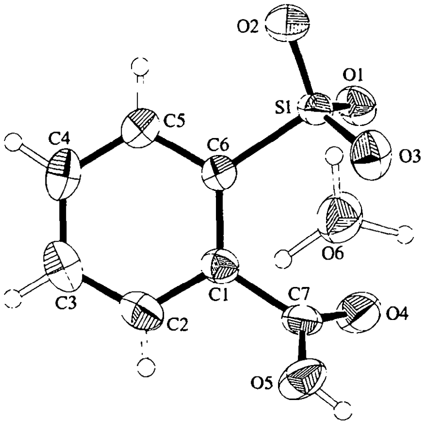


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of hydronium 2-carboxybenzenesulfonate at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.

Experimental

2-Sulfobenzoic acid hydrate ($HO_3SC_6H_4$-2-$CO_2H.xH_2O$), purchased from the Aldrich Chemical Company, was recrystallized from ethanol.

Crystal data

$H_3O^+.C_7H_5O_5S^-$

$M_r = 220.19$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å