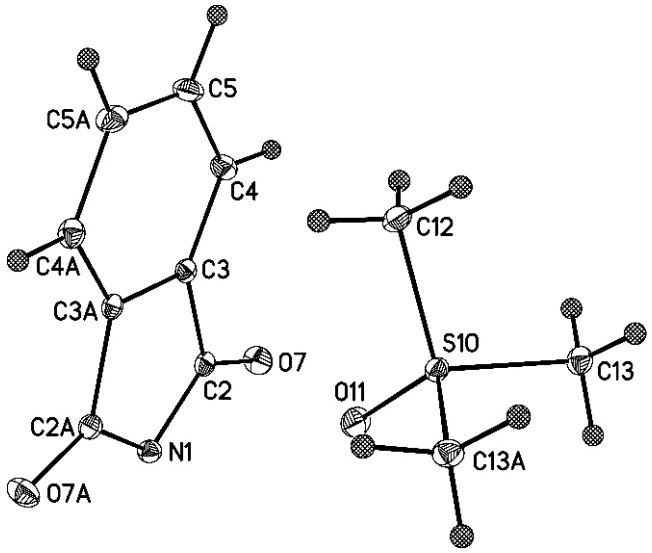
Crystal structure of trimethylsulfoxonium phthalimide, $[C_3H_9SO][C_8H_4NO_2]$

Eyad Mallah[*,I], Qutaiba Abu-Salem[II], Wael Abu Dayyih[I], Mohammed Hamad[I], Manfred Steimann[III] and Caecilia Maichle-Mössmer[III]

[I] Petra University, Faculty of Pharmacy and Medical Sciences, P. O. Box 961343, Amman 11196, Jordan
[II] University of Al al-Bayt, Faculty of Science, Department of Chemistry, Al-Mafraq, Jordan
[III] Institut für Anorganische Chemie der Universität Tübingen, Auf der Morgenstelle 18, 72076 Tübingen, Germany

Received February 21, 2011, accepted and available on-line June 17, 2011; CCDC no. 1267/3411



Abstract

$C_{11}H_{13}NO_3S$, orthorhombic, *Pnma* (no. 62), $a = 8.445(1)$ Å, $b = 10.958(1)$ Å, $c = 12.084(2)$ Å, $V = 1118.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.054$, $wR_{ref}(F^2) = 0.108$, $T = 173$ K.

Source of material

Trimethylsulfoxonium iodide (0.250 g, 1.14 mmol) was added at room temperature. To a solution containing thallium phthalimide (0.397 g, 1.13 mmol) in methanol (30 ml). After stirring overnight, the filtered solution was evaporated to dryness, and the resulting solid was washed with Et_2O (yield 0.220 g, 35 %). The title compound was recrystallized from methanol/Et_2O as colorless crystals.

Discussion

Phthalimide [1(*H*)-isoindoline-1,3(2*H*)-dione] and its derivatives are very important compounds. They are used in the synthesis of antimicrobal activity, as anti drogens and agents for treating tumour necrosis factor. Certain phthalimide derivatives are used as herbicides and for reducing bacterial contamination. Phthalimide forms salts with potassium and thallium due to its high acidity caused by electrophilic carbonyl groups attached to the nitrogen atom. Potassium phthalimide is used in the Gabriel synthesis of primary amines and by reacting with thallium nitrate in aqueous solution produces thallium phthalimide which can give many phthalimide derivatives by reacting with salts containing iodide.

* Correspondence author (e-mail: eyad782002@yahoo.com)

The resulted salt crystallizes with one molecular anion and one cation in the symmetric unit. In the crystal structure, the bond lengths and angles of the anion are quite similar to those reported for potassium phthalimide structure [1].

The bonds are most significantly different in the region near N atom which is the deprotonation site. As a result of deprotonation the internal ring angle at the nitrogen atom in the protonated neutral molecule closes from 112.8(2)° [2] to 107.9(3)° (N1) in its anion. The opening of each of the internal ring angles at the adjacent carbon atoms C2 and C2A by 4 - 5° over-compensates for the closing of the angle at N1 by 4.9°. This suggests that all three angular changes are a consequence of the deprotonation. In addition the C—O bond length [1.231(3) Å] is slightly increased by about 0.03 Å and C—N bond length [1.369(3) Å] is slightly decreased by 0.01 Å. These results and the symmetry about N1 indicate also a symmetric p-electron distribution in the sense of an effective delocalization within the ring. There are no hydrogen interactions in the structure and it is interesting to note that the three S—C bond lengths in the cation are similar (2× 1.750(3) Å and 1× 1.753(4) Å), and the three O–S–C bond angles are slightly similar (2× 112.85(11)° and 113.06(18)°). The molecular anion is planar; the two carbonyl groups are not displaced from the mean plane of their attached molecular anion ring.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.05 × 0.05 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.80 cm^{-1}
Diffractometer, scan mode:	STOE IPDS 2, φ
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15073, 1201
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1048
$N(param)_{refined}$:	107
Programs:	SHELXS-97, SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(4)	8*d*	0.545(4)	0.536(3)	0.645(3)	0.040(9)
H(5)	8*d*	0.401(4)	0.644(3)	0.781(3)	0.05(1)
H(12A)	8*d*	0.210(4)	0.678(3)	0.552(3)	0.037(9)
H(12B)	4*c*	0.055(6)	¾	0.522(4)	0.04(1)
H(13A)	8*d*	0.022(4)	0.634(3)	0.330(2)	0.024(7)
H(13B)	8*d*	0.170(4)	0.620(3)	0.253(3)	0.044(9)
H(13C)	8*d*	0.169(4)	0.552(3)	0.366(3)	0.044(9)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*c*	0.7896(4)	¾	0.4195(2)	0.022(2)	0.023(2)	0.020(1)	0	0.002(1)	0
C(2)	8*d*	0.7336(3)	0.6490(2)	0.4734(2)	0.018(1)	0.024(1)	0.021(1)	–0.001(1)	–0.004(1)	–0.002(1)
C(3)	8*d*	0.6320(3)	0.6865(2)	0.5700(2)	0.016(1)	0.022(1)	0.019(1)	–0.000(1)	–0.0047(9)	0.002(1)
C(4)	8*d*	0.5486(3)	0.6211(3)	0.6477(2)	0.025(1)	0.025(1)	0.028(1)	–0.001(1)	0.000(1)	0.004(1)
C(5)	8*d*	0.4656(3)	0.6867(3)	0.7276(2)	0.025(1)	0.039(2)	0.024(1)	–0.004(1)	0.006(1)	0.006(1)
O(7)	8*d*	0.7625(2)	0.5425(2)	0.4478(2)	0.040(1)	0.022(1)	0.038(1)	0.0030(9)	0.0078(9)	–0.0047(8)
S(10)	4*c*	0.2239(1)	¾	0.38169(7)	0.0171(4)	0.0212(4)	0.0164(4)	0	–0.0016(3)	0
O(11)	4*c*	0.3936(3)	¾	0.3654(2)	0.018(1)	0.048(2)	0.027(2)	0	–0.001(1)	0
C(12)	4*c*	0.1693(5)	¾	0.5217(3)	0.029(2)	0.026(2)	0.017(2)	0	–0.002(2)	0
C(13)	8*d*	0.1314(3)	0.6217(3)	0.3246(2)	0.028(2)	0.020(1)	0.023(1)	–0.001(1)	–0.001(1)	–0.004(1)

Acknowledgment. X-ray data were collected at the University of Tübingen using a Stoe IPDS 2 diffractometer, we would like to acknowledge the Deanship of Scientific Research at Petra University for their support and kind cooperation.

References

1. Nagel, N.; Bock, H.; Bats, J. W.: Potassium Phthalimide. Acta Crystallogr. **C52** (1996) 1344.
2. Ng, S. W.: Structure of 1*H*-isoindole-1,3(2*H*)-dione (Phthalimide). Acta Crystallogr. **C48** (1992) 1694-1695.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.