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Crystal structure of pentamethylpyrylium triiodide, $[(CH_3)_5C_5O][I_3]$

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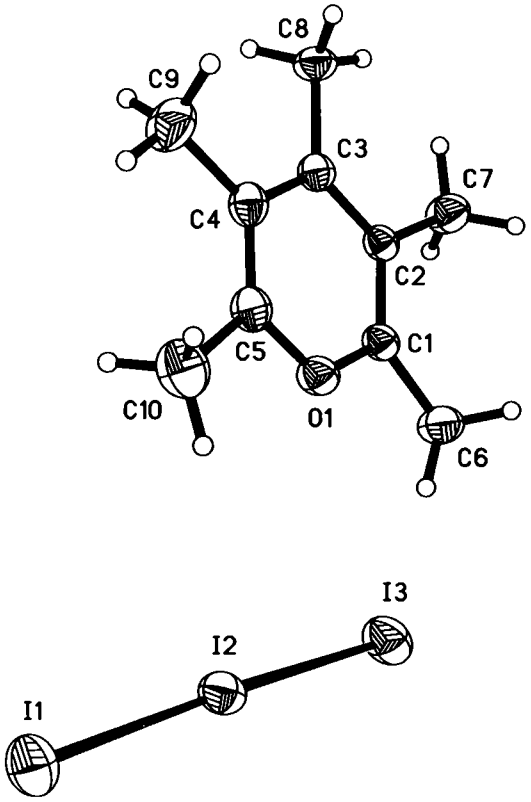

Abstract

$C_{10}H_{15}I_3O$, orthorhombic, *Pnma* (no. 62), a = 13.750(3) Å, b = 7.288(2) Å, c = 15.140(3) Å, V = 1517.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.071, T = 150 K.

Source of material

The title compound was isolated as a by-product of the reaction of methylmagnesium iodide with 2,3,4,5-tetramethylcyclopenta-2-en-1-one in diethyl ether.

Discussion

The pyrylium salts represent one of the fundamental heterocyclic systems and are the basis of important natural products such as anthocyanin [1]. They function as very useful organic precursor due to their strong electrophilic reactivity toward many other nucleophiles [2,3]. The pyrylium ring is exactly planar. The two C—O bonds have identical lengths within their standard errors. The C—C bond lengths split up into two sets. The two C—C bonds adjacent to C—O bonds are shorter (1.361(10) Å and 1.371(9) Å) than the two others ones (1.399(9) Å and 1.419(10) Å). The C–O–C and the two O–C–C bond angles (range: 120.6(6)° – 121.7(6)°) are slightly larger than 120°, whereas the C–C–C bond angles (range: 118.3(7)° – 119.5(6)°) are slightly smaller. The form of the pyrylium ring is comparable to those of the pyrylium ring in the known salt 2,6-diphenyl-4-carboxypyrylium perchlorate [4]. The C—C bond lengths to the methyl groups range from 1.48(1) Å to 1.53(1) Å, giving an average of 1.506 Å. The I—I bond lengths in the triiodide anion are 2.9347(8) Å for I1—I2 and 2.9166(8) Å for I2—I3. The arrangement of the three iodine atoms is not exactly linear. The I–I–I bond angle is 177.63(2)°. This values are comparable to those in known salts of the composition R_4PI_3, R_4AsI_3 and R_4SbI_3 (R = alkyl) [5].

Table 1. Data collection and handling.

Crystal:	brown column, size 0.18 × 0.21 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	61.55 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω/φ
$2\theta_{max}$:	53.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22409, 1768
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1571
$N(param)_{refined}$:	85
Programs:	SHELXS-97 [6], SHELXL-97 [7], DIAMOND [8]

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

Atom	Site	x	y	z	U_{iso}
H(6A)	4*c*	0.4731	¼	–0.0079	0.054
H(6B)	8*d*	0.4848	0.3576	–0.0970	0.054
H(7A)	4*c*	0.3095	¼	–0.2948	0.054
H(7B)	8*d*	0.3951	0.1424	–0.2500	0.054
H(8A)	4*c*	0.1823	¼	–0.2892	0.045
H(8B)	8*d*	0.0989	0.3576	–0.2412	0.045
H(9A)	4*c*	0.0215	¼	–0.0055	0.067
H(9B)	8*d*	0.0157	0.1424	–0.0951	0.067
H(10A)	4*c*	0.2288	¼	0.1348	0.071
H(10B)	8*d*	0.1346	0.1424	0.1077	0.071

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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}
I(1)	4c	0.22707(4)	¼	0.49503(4)	0.0435(3)	0.0365(3)	0.0370(3)	0	0.0085(2)	0
I(2)	4c	0.36095(4)	¼	0.34407(3)	0.0376(3)	0.0269(2)	0.0270(2)	0	–0.0044(2)	0
I(3)	4c	0.50072(4)	¼	0.19917(3)	0.0500(3)	0.0419(3)	0.0274(3)	0	0.0033(2)	0
O(1)	4c	0.3039(4)	¼	–0.0032(3)	0.030(3)	0.035(3)	0.023(2)	0	0.000(2)	0
C(1)	4c	0.3506(5)	¼	–0.0814(4)	0.028(3)	0.024(3)	0.021(3)	0	0.000(3)	0
C(2)	4c	0.2994(5)	¼	–0.1591(4)	0.027(3)	0.021(3)	0.020(3)	0	0.000(3)	0
C(3)	4c	0.1977(5)	¼	–0.1556(5)	0.027(3)	0.021(3)	0.028(3)	0	–0.002(3)	0
C(4)	4c	0.1503(5)	¼	–0.0723(5)	0.028(4)	0.027(3)	0.030(4)	0	0.004(3)	0
C(5)	4c	0.2062(5)	¼	0.0019(5)	0.030(4)	0.031(3)	0.028(4)	0	0.005(3)	0
C(6)	4c	0.4578(6)	¼	–0.0698(5)	0.032(4)	0.045(4)	0.030(4)	0	–0.005(3)	0
C(7)	4c	0.3549(5)	¼	–0.2466(5)	0.028(4)	0.047(4)	0.032(4)	0	0.000(3)	0
C(8)	4c	0.1392(5)	¼	–0.2394(5)	0.029(4)	0.036(4)	0.025(3)	0	–0.007(3)	0
C(9)	4c	0.0408(6)	¼	–0.0665(6)	0.028(4)	0.056(5)	0.049(5)	0	0.007(4)	0
C(10)	4c	0.1731(7)	¼	0.0966(6)	0.050(5)	0.059(5)	0.034(4)	0	0.012(4)	0

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