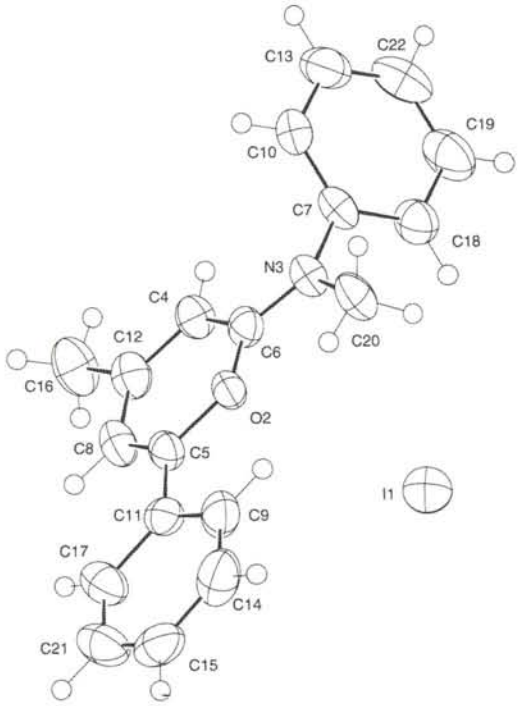
Crystal structure of *N*-(4-methyl-6-phenyl-2*H*-pyran-2-ylidene)-*N*-methyl benzenaminium iodide, $C_{19}H_{18}INO$

R. Kakou-Yao[I], C. Uncuta[II] and J. P. Aycard*,[III]

[I] Université de Cocody, Laboratoire de Cristallographie et de Physique Moléculaire, UFR SSMT, 22 BP 582 Abidjan 22, Côte d'Ivoire
[II] Centre de Chimie Organique, "C. D. Nenitzescu", 71141 Bucharest 15-258, Roumania
[III] Faculté des Sciences et Techniques de Saint Jérôme, Laboratoire de Spectrométries et Dynamique Moléculaire, Case 542 Avenue Escadrille Normandie Niemen, F-13397 Marseille Cedex 20, France

Received March 14, 2000, CCDC-No. 1267/423



Abstract

$C_{19}H_{18}INO$, orthorhombic, *Pbca* (No. 61), $a = 8.6733(1)$ Å, $b = 16.9360(1)$ Å, $c = 23.7983(1)$ Å, $V = 3495.8$ Å^3, $Z = 8$, $R_{gt}(F) = 0.053$, $wR_{ref}(F) = 0.082$, $T = 293$ K.

Source of material

On heating gently the imine with excess of methyl iodide, the compound was obtained. The methode using is explained by Uncuta et al. [1].

Discussion

For the study of pyrylium salts and hydroxylamine, many reactions mechanisms were presented. Among these reactions appear the compound we make here the structure. The results of the single crystal analysis of the compound were in full agreement with configurational assignment in solution. We note the distances N3—C6 1.329(4) Å; N3—C7 1.438(4) Å; N3—C20 1.460(4) Å; O2—C5 1.380(3) Å; O2—C6 1.329(4) Å; C5—C11 1.465(4) Å; C12—C16 1.487(5) Å. The P_1(O2C5C8C12C4C6) plane forms an angle of 107.40(3)° with P_2(C7C18C19C22C13C10) plane and an angle of 2.70(3)° with P_3(O2C5C8C12C4C6) plane.

Table 1. Data collection and handling.

Crystal:	orange plate, size 0.03 × 0.32 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	18.34 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, φ
$2\theta_{max}$:	50.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3847, 3464
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 2714
$N(param)_{refined}$:	199
Programs:	MAXUS [2], MULTAN [3], ORTEPII [4]

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

Atom	Site	x	y	z	U_{iso}
H(4)	8*c*	0.13754	0.11010	0.80851	0.05
H(8)	8*c*	–0.10319	–0.07805	0.86743	0.05
H(9)	8*c*	0.23552	–0.04798	1.01027	0.05
H(10)	8*c*	0.20705	0.25294	0.86917	0.05
H(13)	8*c*	0.30425	0.34319	0.80334	0.05
H(14)	8*c*	0.22224	–0.13514	1.08616	0.05
H(15)	8*c*	0.04689	–0.24206	1.08408	0.05
H(16A)	8*c*	–0.06639	0.06165	0.75523	0.05
H(16B)	8*c*	–0.20309	0.02825	0.79113	0.05
H(16C)	8*c*	–0.09069	–0.02985	0.76023	0.05
H(17)	8*c*	–0.12252	–0.16313	0.93655	0.05
H(18)	8*c*	0.57566	0.10833	0.84119	0.05
H(19)	8*c*	0.67857	0.20320	0.77961	0.05
H(20A)	8*c*	0.35797	0.06377	0.97449	0.05
H(20B)	8*c*	0.41387	0.15177	0.97239	0.05
H(20C)	8*c*	0.51167	0.08537	0.94339	0.05
H(21)	8*c*	–0.13064	–0.24919	1.01264	0.05
H(22)	8*c*	0.53879	0.31777	0.75782	0.05

* Correspondence author (e-mail: aycard@piimsdm3.univ-mrs.fr)

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)	8*c*	0.53328(3)	–0.08626(2)	0.86943(1)	0.0628(2)	0.0557(2)	0.0803(2)	–0.0010(1)	–0.0012(1)	0.0091(1)
O(2)	8*c*	0.1779(2)	0.0148(1)	0.92584(8)	0.031(1)	0.038(1)	0.041(1)	–0.0058(8)	0.0001(8)	0.0003(9)
N(3)	8*c*	0.3239(3)	0.1160(2)	0.8999(1)	0.036(1)	0.045(1)	0.045(1)	–0.010(1)	0.000(1)	–0.001(1)
C(4)	8*c*	0.1189(4)	0.0712(2)	0.8370(1)	0.040(2)	0.048(2)	0.041(2)	–0.005(1)	0.003(1)	0.001(1)
C(5)	8*c*	0.0640(3)	–0.0412(2)	0.9186(1)	0.033(2)	0.039(2)	0.048(2)	–0.004(1)	0.010(1)	–0.004(1)
C(6)	8*c*	0.2089(3)	0.0677(2)	0.8862(1)	0.032(2)	0.042(2)	0.041(2)	0.001(1)	0.006(1)	–0.003(1)
C(7)	8*c*	0.3798(4)	0.1729(2)	0.8600(1)	0.043(2)	0.045(2)	0.044(2)	–0.016(1)	0.000(1)	–0.004(1)
C(8)	8*c*	–0.0223(4)	–0.0399(2)	0.8721(1)	0.037(2)	0.054(2)	0.048(2)	–0.011(1)	0.001(2)	–0.009(1)
C(9)	8*c*	0.1594(4)	–0.0892(2)	1.0109(2)	0.038(2)	0.058(2)	0.066(2)	0.003(2)	0.006(2)	0.016(2)
C(10)	8*c*	0.3013(4)	0.2418(2)	0.8496(2)	0.035(2)	0.053(2)	0.068(2)	–0.005(2)	–0.004(2)	0.002(2)
C(11)	8*c*	0.0582(3)	–0.0961(2)	0.9661(1)	0.041(2)	0.036(2)	0.048(2)	0.004(1)	0.013(1)	–0.001(1)
C(12)	8*c*	0.0029(3)	0.0172(2)	0.8302(1)	0.041(2)	0.056(2)	0.039(2)	–0.003(1)	0.004(1)	–0.006(1)
C(13)	8*c*	0.3604(5)	0.2958(2)	0.8116(2)	0.069(3)	0.044(2)	0.075(3)	–0.012(2)	–0.012(2)	0.007(2)
C(14)	8*c*	0.1545(4)	–0.1417(3)	1.0546(2)	0.049(2)	0.088(3)	0.065(2)	0.012(2)	0.003(2)	0.021(2)
C(15)	8*c*	0.0465(5)	–0.2036(2)	1.0545(2)	0.067(3)	0.052(2)	0.080(3)	0.018(2)	0.031(2)	0.022(2)
C(16)	8*c*	–0.0983(4)	0.0196(3)	0.7797(2)	0.057(2)	0.088(3)	0.047(2)	–0.019(2)	–0.006(2)	–0.003(2)
C(17)	8*c*	–0.0509(4)	–0.1568(2)	0.9669(2)	0.060(2)	0.047(2)	0.060(2)	–0.012(2)	0.009(2)	–0.010(2)
C(18)	8*c*	0.5202(4)	0.1562(2)	0.8338(2)	0.052(2)	0.051(2)	0.058(2)	–0.004(2)	0.013(2)	–0.004(2)
C(19)	8*c*	0.5806(5)	0.2127(3)	0.7971(2)	0.069(3)	0.068(3)	0.071(3)	–0.020(2)	0.027(2)	0.001(2)
C(20)	8*c*	0.4091(4)	0.1029(2)	0.9520(2)	0.048(2)	0.060(2)	0.051(2)	–0.021(2)	–0.009(2)	0.002(2)
C(21)	8*c*	–0.0540(5)	–0.2083(2)	1.0111(2)	0.074(3)	0.046(2)	0.072(3)	–0.013(2)	0.029(2)	–0.001(2)
C(22)	8*c*	0.4985(5)	0.2807(2)	0.7845(2)	0.094(3)	0.057(2)	0.063(2)	–0.032(2)	0.006(2)	0.008(2)

Acknowledgment. We thank the French government for financial support.

References

1. Uncuta, C.; Tudose, A.; Caproiu, M. T.; Plaveti, M.; Kakou-Yao, R.: Beckmann and Cyclization Reactions of δ-Oxo-α,β-Unsaturated Ketoximes Obtained from Pyrylium Salts and Hydroxylamine. Formation of 2-Aryl(or Akyl)amino-4,6 disubstituted Pyrylium Salts. Tetrahedron **55** (1999) 15011-15024.
2. Mackay, S.; Gilmore, C. J.; Edwards, C.; Tremayne, M.; Stewart, N.; Shankland, K.: University of Glasgow, Scotland, UK, Nonius BV, Delft, The Netherlands and MacScience Co. Ltd., Yokohama, Japan1998.
3. Main, P.; Fiske, S. J.; Hull, S. E.; Lessinger, L.; Germain, G.; Declercq, J. P.; Woolfson, M. M.: MULTAN 11/82. A system of Computer Programs for the automatic Solution of Crystal Structures from X-ray Diffraction Data. Universities of York, England and Louvain, Belgium 1982.
4. Johnson, C. K.: ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA 1976.