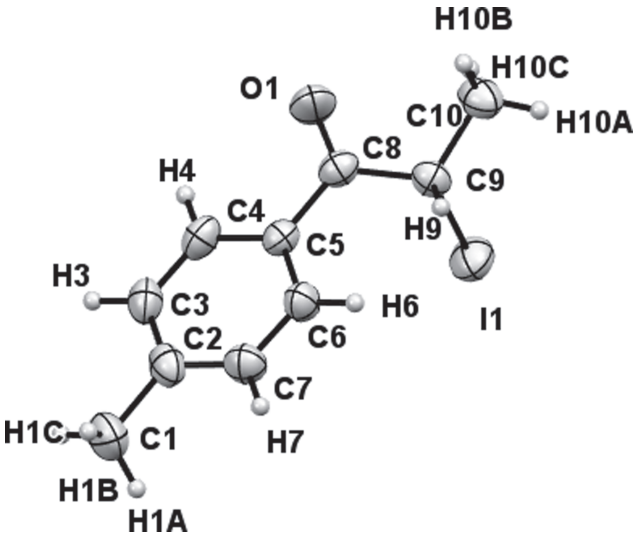
Hui Liu, Linglan Li*, Shu Zhang and Junfeng Miao

The Crystal structure of 2-iodo-1-(*p*-tolyl)propan-1-one, $C_{10}H_{11}IO$



https://doi.org/10.1515/ncrs-2017-0264
Received September 4, 2017; accepted December 12, 2017; available online January 15, 2018

Abstract

$C_{10}H_{11}IO$, monoclinic, $P2_1/c$ (no. 14), $a = 6.5799(5)$ Å, $b = 10.1508(9)$ Å, $c = 15.0983(12)$ Å, $\beta = 92.313(7)°$, $V = 1007.61(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0343$, $wR_{ref}(F^2) = 0.0860$, $T = 297$ K.

CCDC no.: 1565953

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

Following a literature procedure [4, 5], 0.21 g powdered CuO and 0.65 g I_2 were added to 20 mL methanol solvent containing 0.68 g 1-(*p*-tolyl)ethanone under stirring conditions. Stirring was continued for 5 min and then refluxed for 5 h. After the reaction was complete, the mixture was filtered and the solvent was removed under reduced pressure. The residue was poured into 30 mL 10% $Na_2S_2O_3$ solution. The mixture was extracted with 3×20 mL EtOAc, and the organic layer was dried by Na_2SO_4. After removal of the solvent, the residue was recrystallized giving the target product with 84% yield, M.P.: 107–109 °C. **^{1}HNMR** (400 MHz, $CDCl_3$): 2.08 (d, 3H), 2.42 (s, 3H), 5.47 (q, 1H), 7.26 (d, 2H), 7.92 (d, 2H).

Table 1: Data collection and handling.

Crystal:	Block, clear light yellow
Size:	$0.3 \times 0.3 \times 0.15$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.13 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω-scans
θ_{max}, completeness:	27°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7577, 2189, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1794
$N(param)_{refined}$:	111
Programs:	CrysAlis^{PRO} [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
I1	0.76222(4)	0.64119(3)	0.55963(2)	0.06026(14)
O1	0.9553(5)	0.8332(2)	0.7495(2)	0.0639(8)
C5	0.7090(5)	0.6805(3)	0.7907(2)	0.0346(7)
C8	0.8844(5)	0.7243(3)	0.7395(2)	0.0385(7)
C9	0.9742(5)	0.6333(3)	0.6728(2)	0.0378(7)
H9	0.977974	0.543479	0.696578	0.045*
C6	0.6548(5)	0.5496(3)	0.8001(2)	0.0421(8)
H6	0.730611	0.484121	0.773760	0.051*
C2	0.3728(5)	0.6097(4)	0.8873(2)	0.0450(8)
C4	0.5924(6)	0.7759(3)	0.8312(2)	0.0451(8)
H4	0.627055	0.864378	0.826302	0.054*
C10	1.1840(5)	0.6712(4)	0.6446(3)	0.0527(9)
H10A	1.225047	0.612774	0.598625	0.079*
H10B	1.278652	0.664753	0.694514	0.079*
H10C	1.181680	0.760022	0.622841	0.079*
C7	0.4897(5)	0.5158(4)	0.8483(2)	0.0465(8)
H7	0.456515	0.427325	0.854567	0.056*
C3	0.4262(5)	0.7403(4)	0.8784(2)	0.0467(8)
H3	0.348987	0.805162	0.904651	0.056*
C1	0.1879(6)	0.5685(5)	0.9367(3)	0.0637(11)
H1A	0.107746	0.508170	0.900945	0.096*
H1B	0.107850	0.644799	0.949352	0.096*
H1C	0.230530	0.526310	0.991272	0.096*

***Corresponding author: Linglan Li,** Hubei University, The College of Chemistry and Chemical Engineering, Wuhan 430062, China, e-mail: iamliuhui@live.cn
Hui Liu: Wuhan Institute of Technology, School of Materials Science and Engineering, Wuhan 430205, China
Shu Zhang and Junfeng Miao: Hubei University, The College of Chemistry and Chemical Engineering, Wuhan 430062, China

Experimental details

All hydrogen atoms were placed in idealized positions and refined as riding on their parent atoms.

Comment

α-Iodoketones have received broad attention because of their attractive properties and potential in various applications. For example, their high reactivity makes them available to react with a variety of nucleophiles to synthesize useful compounds, and their biologically active property makes them highly promising for utilization in medicine as drugs or diagnostic aids [6–8].

This paper reports the crystal structure of an α-iodo arylketone, which is only built up by the $C_{10}H_{11}IO$ molecules (*cf*. the figure), in which all bond lengths are in normal ranges. There are no classical hydrogen bonds to connect adjacent molecules.

Acknowledgements: We gratefully acknowledge support by the science foundation of Wuhan institute of technology (No. K201509).

References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.38.41, Agilent Technologies UK Ltd, Oxford, UK (2015).
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i, Crystal Impact, Bonn, Germany (2012).
4. Yin, G.; Zhou, B.; Meng, X.; Wu, A.; Pan, Y.: Efficient C—C double-bond formation reaction *via* a new synthetic strategy: a self-sorting tandem reaction. Org. Lett. **11** (2006) 2245–2248.
5. Yin, G.; Gao, M.; She, N.; Hu, S.; Wu, A.; Pan, Y.: Highly efficient and clean method for direct α-iodination of aromatic ketones. Synthesis **20** (2007) 3113–3116.
6. Lee, J. C.; Bae, Y. H.: Efficient α-iodination of carbonyl compounds under solvent-free conditions using microwave irradiation. Synlett **4** (2003) 507–508.
7. Lee, J. C.; Jin, Y. S.: Efficient method for α-iodination of ketones. Synth. Commun. **29** (1999) 2769–2774.
8. Lee, J. C.; Kim, J.; Park, H. J.; Kwag, B.; Lee, S. B.: Direct metal-free α-iodination of arylketones induced by iodine or iodomethane with HTIB in ionic liquid. Bull. Korean Chem. Soc. **31** (2010) 1385–1386.