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(2,6-Diisopropylphenyl)isopropylidene-ammonium iodide

Robert J. Baker, Helga Bettentrup and Cameron Jones*

Department of Chemistry, Cardiff University, PO Box 912, Cardiff CF10 3TB, Wales

Correspondence e-mail: jonesca6@cardiff.ac.uk

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Key indicators

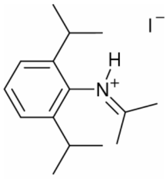
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.047
wR factor = 0.099
Data-to-parameter ratio = 18.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{14}H_{24}N^+\cdot I^-$, was formed by a Schiff base condensation of 2,6-diisopropylaniline and acetone, using GaI as a Lewis acid. A strong interaction from the iminium hydrogen N—H to the iodide counter-ion is observed. The ^{1}H and ^{13}C NMR data are also reported.

Comment

We have been investigating the reactivity of GaI (Green *et al.*, 1990) in C—C coupling reactions (Baker & Jones, 2003) and in the preparation of Ga^{I} N-heterocyclic carbene analogues (Baker *et al.*, 2002).



(I)

In the course of our investigations, we have found that GaI can be used as a Lewis acid catalyst for the Schiff base condensation of a primary amine and a ketone. The Schiff base condensation reaction of an aldehyde or ketone with an amine is well known to be catalysed by Lewis acids (Armesto *et al.*, 1986). The addition of 2,6-diisopropylaniline to a suspension of GaI in toluene, followed by half an equivalent of acetone, gives rise to the expected condensation product. This presumably reacts further with HI to give the title product, (I), in good isolated yields.

The crystal structure of this salt displays a C1—N1 bond length in the expected range for compounds of this type (Scholz *et al.*, 1993). There is a close contact which may be regarded as an N—H···I hydrogen bond [N1—H1 = 0.870 (19), H1···I1 2.57 (2), N1···I1 3.423 (4) Å and N1—H1···I1 167 (4)°].

Experimental

To a solution of GaI (2.07 mmol) in toluene (10 ml) was added a solution of 2,6-diisopropylaniline (0.37 ml, 1.96 mmol) in toluene (10 ml), followed by a solution of acetone (0.07 ml, 1.02 mmol) in toluene (10 ml). After stirring for 4 h under argon, the solvent was removed under vacuum and the residue extracted with CH_2Cl_2 (20 ml). Concentration and cooling to 243 K afforded colourless blocks of the title compound (0.08 g, 23%, m.p. 414–417 K). IR (Nujol): ν_{max} 2965, 2664, 1935, 1835, 1724, 1654, 1589, 1558, 1460, 1373, 1252, 1167, 1076, 1046, 971, 931, 825, 793, 722 cm^{-1}. ^{1}H NMR [400 MHz, CD_2Cl_2]: δ 1.13 (*d*, 6H, *J* = 6.89 Hz, CH_3), 1.20 (*d*, 6H, *J* = 6.82 Hz, CH_3), 2.14 (*s*, 3H, $NCCH_3$), 2.71 (*sept*, 2H, *J* = 6.79 Hz, CH), 3.04 (*s*, 3H, NCCH$_3$), 7.26 (*d*, 2H, J = 7.78, *m*-Ar), 7.42 (*t*, 1H, *J* = 11.18 Hz, *p*-Ar). ^{13}C NMR [100 MHz, CD$_2$Cl$_2$]: δ 22.6 (CH$_3$), 23.7 ((CH$_3$)$_2$CN), 24.7 (CH$_3$), 24.9 [(CH$_3$)$_2$CN], 29.1 [CH(Me)$_2$], 124.8 (*m*-Ar), 129.8 (*o*-Ar), 131.1 (*p*-Ar), 143.1 (*i*-Ar), 193.9 (C—N). MS (APCI): *m*/*z* 218.0 (M^+, 100%), 201.9 (M^+–NH$_2$), 175.8 (M^+–HC(Me)$_2$).

Crystal data

$C_{15}H_{24}N^+\cdot I^-$
$M_r = 345.25$
Monoclinic, $P2_1/c$
$a = 13.426$ (3) Å
$b = 11.128$ (2) Å
$c = 12.211$ (2) Å
$\beta = 113.28$ (3)°
$V = 1675.8$ (6) Å^3
$Z = 4$
$D_x = 1.368$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8761 reflections
$\theta = 2.9$–25.0°
$\mu = 1.89$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.594$, $T_{\max} = 0.598$
8952 measured reflections
2944 independent reflections
2159 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\max} = 25.1°$
$h = -15 \rightarrow 15$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.099$
$S = 1.07$
2944 reflections
164 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0144P)^2 + 2.3024P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.69$ e Å^{-3}
$\Delta\rho_{\min} = -0.86$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C13	1.281 (5)	C6—C10	1.518 (6)
N1—C1	1.447 (5)	C13—C14	1.467 (7)
N1—H1	0.870 (19)	C13—C15	1.480 (6)
C2—C7	1.527 (6)		
C13—N1—C1	127.2 (4)	C6—C1—N1	118.0 (4)
C13—N1—H1	115 (3)	N1—C13—C14	121.1 (4)
C1—N1—H1	117 (3)	N1—C13—C15	119.8 (4)
C2—C1—N1	118.8 (4)		

Atom H1, attached to N1, was refined isotropically, with a restrained N—H bond length. All other H atoms were positioned geometrically and refined with riding-model constraints.

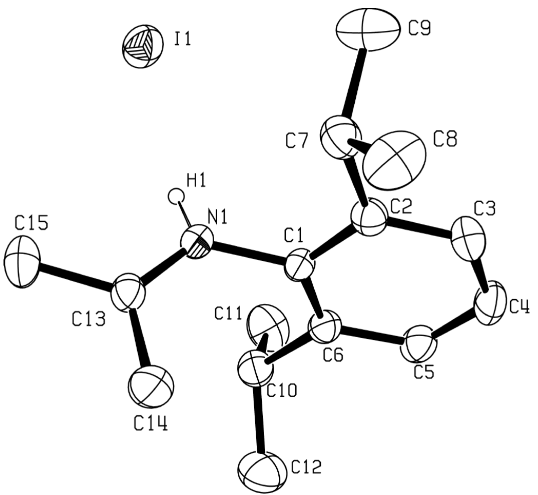


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 25% probability level.

Data collection: *COLLECT* (Hooft, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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