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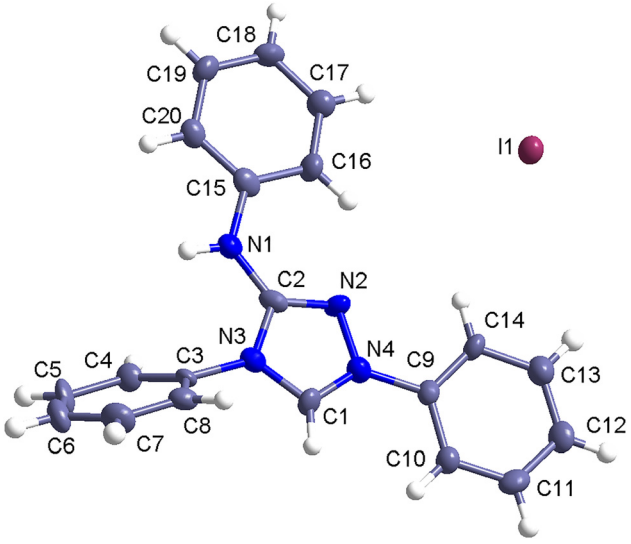
The crystal structure of 3-anilino-1,4-diphenyl-4*H*-1,2,4-triazol-1-ium iodide, $C_{20}H_{17}N_4I$



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Abstract

$C_{20}H_{17}N_4I$, monoclinic, $P2_1/n$ (no. 14), a = 7.304(3) Å, b = 14.830(7) Å, c = 16.482(7) Å, β = 98.202(12)°, V = 1767.2(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0568, $wR_{ref}(F^2)$ = 0.1359, T = 150 K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

Nitron (0.5 g, 1.60 mmol) in THF (15 mL) was placed in a Schlenk flask, to which a solution of HI (57% w/w, 0.36 g, 1.60 mmol) in THF (5 mL) was added dropwise. The mixture was allowed to stir at room temperature for 10 min and the white solid formed was collected on a frit and dried under vacuum. Crystals suitable for the structural determination was obtained from slow evaporation of a methanol solution containing the compound.

Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.82 mm^{-1}
Diffractometer, scan mode:	Bruker Apex-II, φ and ω-scans
θ_{max}, completeness:	27°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,053, 3769, 0.113
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2141
$N(param)_{refined}$:	227
Programs:	Bruker programs [1], SHELX [2], DIAMOND [3]

Experimental details

All H atoms were fixed at calculated positions and then riding refinements were performed. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

Comment

Nitron is a commercial name to 1,4-diphenyl-3-(phenylamino)-1*H*-1,2,4-triazolium inner salt, which has been extensively used in diverse fields for decades [4–8]. Recently, nitron has been shown to be a reliable *N*-heterocyclic carbene (NHC) precursor for obtaining a wide range of transition-metal NHC complexes [9–11]. In the course of our investigation on metal NHC complexes, we had obtained its hydroiodide salt as a by-product. Crystal structures for nitron and its non-stoichiometric hydrochloride salt have been published [12]. But the structure of its stoichiometric hydroiodide salt has not yet been described.

The title compound crystallizes in the monoclinic space group $P2_1/n$. Among the four C–N bond distances in

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.56872 (8)	0.12954 (3)	0.75290 (3)	0.0299 (2)
N2	0.1730 (9)	0.0904 (4)	0.4935 (3)	0.0250 (15)
C9	0.0807 (10)	0.2444 (5)	0.5183 (4)	0.0232 (17)
C15	0.3227 (11)	−0.0959 (5)	0.4937 (4)	0.0293 (19)
C17	0.4414 (11)	−0.1190 (5)	0.6349 (4)	0.0312 (19)
H17	0.4647	−0.0966	0.6894	0.037*
N1	0.2326 (8)	−0.0457 (4)	0.4259 (3)	0.0281 (16)
H1N	0.2179	−0.0724	0.3777	0.034*
C16	0.3557 (11)	−0.0633 (5)	0.5737 (4)	0.032 (2)
H16	0.3199	−0.0038	0.5860	0.038*
N3	0.0885 (8)	0.0840 (4)	0.3591 (3)	0.0241 (15)
N4	0.0970 (9)	0.1712 (4)	0.4628 (3)	0.0273 (16)
C10	−0.0140 (11)	0.3222 (5)	0.4889 (4)	0.0280 (19)
H10	−0.0659	0.3270	0.4329	0.034*
C7	0.0638 (12)	0.0703 (6)	0.1337 (5)	0.040 (2)
H7	0.1034	0.1031	0.0898	0.048*
C4	−0.0590 (11)	−0.0232 (5)	0.2600 (4)	0.0293 (19)
H4	−0.1027	−0.0546	0.3037	0.035*
C12	0.0420 (11)	0.3832 (6)	0.6263 (4)	0.033 (2)
H12	0.0255	0.4300	0.6639	0.040*
C3	0.0477 (10)	0.0514 (5)	0.2759 (4)	0.0216 (17)
C18	0.4942 (11)	−0.2060 (5)	0.6197 (4)	0.0298 (19)
H18	0.5487	−0.2438	0.6631	0.036*
C5	−0.1044 (12)	−0.0539 (6)	0.1801 (4)	0.037 (2)
H5	−0.1791	−0.1061	0.1690	0.045*
C8	0.1115 (11)	0.1017 (5)	0.2139 (4)	0.0301 (19)
H8	0.1838	0.1546	0.2256	0.036*
C13	0.1367 (12)	0.3065 (6)	0.6523 (5)	0.040 (2)
H13	0.1880	0.3009	0.7084	0.048*
C2	0.1674 (11)	0.0397 (5)	0.4289 (4)	0.0249 (18)
C1	0.0486 (11)	0.1678 (5)	0.3832 (4)	0.0268 (19)
H1C	−0.0045	0.2153	0.3489	0.032*
C11	−0.0307 (11)	0.3916 (5)	0.5427 (5)	0.034 (2)
H11	−0.0917	0.4457	0.5233	0.041*
C14	0.1597 (11)	0.2362 (5)	0.5983 (4)	0.0275 (19)
H14	0.2286	0.1840	0.6168	0.033*
C19	0.4658 (11)	−0.2368 (5)	0.5398 (4)	0.032 (2)
H19	0.5086	−0.2951	0.5278	0.038*
C6	−0.0402 (12)	−0.0077 (6)	0.1166 (4)	0.036 (2)
H6	−0.0671	−0.0292	0.0619	0.043*
C20	0.3755 (11)	−0.1839 (5)	0.4765 (4)	0.0274 (19)
H20	0.3500	−0.2072	0.4223	0.033*

the triazolium ring, the C2–N2 distance of 1.300(9) Å is the shortest, whereas the longest one is the C2–N3 distance of 1.377(9) Å. The two C–N distances flanking the C1 carbon are 1.349(9) and 1.311(8) Å, respectively. The N–N distance is 1.386(8) Å. The least squares plane of the phenyl ring at the four-position was twisted from that of the triazolium ring with an inter-planar angle of 42.9(3)°. The other two phenyl rings are, however, almost co-planar with the heterocyclic ring. The planarity of these rings allows intermolecular π–π stacking interactions to occur. The iodide ions are involved in short contacts of the types N–H···I and C–H···I with the organic cations, linking them into one-dimensional zigzag chains along the *b*-axis. The structure is further stabilized by π–π stacking interactions between these chains.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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