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9-*p*-Toluidinoanthracene

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Condensation of anthrone with *p*-toluidine affords 9-*p*-toluidinoanthracene, $C_{21}H_{17}N$. The aromatic ring systems enclose a dihedral angle of 79.97 (3)°. One short intermolecular contact from the amino H atom is found to an aromatic C atom can be found.

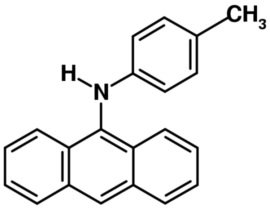
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.140
Data-to-parameter ratio = 21.8

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

Comment

Anthracene derivatives have been widely studied because of their important biological and photophysical properties (Seonkyung *et al.*, 1997; Lahlou *et al.*, 1998). In our research, we concentrated on alkyl and aryl anthracene derivatives. Recently, we described the preparation of diethyl 2-anthracen-9-ylmethylenemalonate (Elazami *et al.*, 1999). In this paper we report on the synthesis and crystal structure of a new aryl-anthracene, incorporating nitrogen in the chain, *N*-(9-anthracene) *p*-toluidine, which was prepared by the condensation reaction of anthrone and *p*-toluidine. The title compound (I) has a high stability at room temperature [confirmed by mass spectroscopy 283 (M^+, 100%)]. Since NMR and mass spectroscopy did not provide sufficient information about the conformation of the reaction product, we have carried out the X-ray structure analysis.



(I)

Bond lengths and angles are in the usual ranges. The N atom deviates only slightly from the plane of the anthracene ring system [0.036 (2) Å] and from the plane of the tolyl ring [0.075 (2) Å]. Both aromatic ring systems enclose a dihedral angle of 79.97 (3)°. In the absence of a classical hydrogen bond acceptor, only one intermolecular N—H···C contact less than 3 Å can be found.

Experimental

In a 100 ml three-necked flask fitted with a reflux condenser were placed anthrone (5 g, 25 mmol), *p*-toluidine (8 g, 075 mmol), which had previously been dissolved in benzene (40 ml), and dichloromethane (50 ml). The mixture was stirred at room temperature. Then aluminium trichloride (1 ml), dissolved in ethyl ether (20 ml), was added. At the end of the addition, the mixture was stirred and refluxed for 24 h. The solution was extracted with dichloromethane and dried with anhydrous sodium sulfate. The solvent was removed on a rotary evaporator and the residue was purified by flash column

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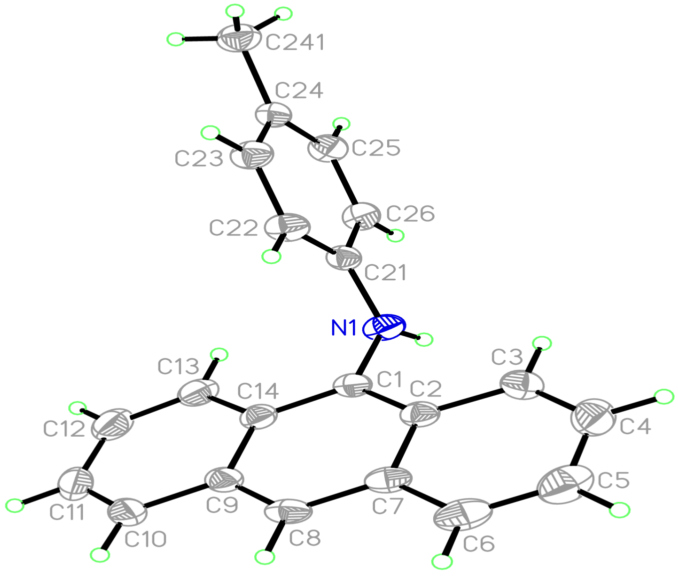

Figure 1
A perspective view of the title compound with the atom-numbering scheme. Displacement ellipsoids are at the 50% probability level.

chromatography over silica (hexane). Recrystallization from ethyl ether/hexane (volume ratio: 3/1) led to yellow plate crystals.

Crystal data

$C_{21}H_{17}N$
$M_r = 283.36$
Monoclinic, $P2_1/n$
$a = 10.367$ (1) Å
$b = 6.343$ (1) Å
$c = 23.361$ (2) Å
$\beta = 100.98$ (1)°
$V = 1508.0$ (3) Å^3
$Z = 4$

$D_x = 1.248$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6696 reflections
$\theta = 2$–25°
$\mu = 0.07$ mm^{-1}
$T = 173$ (2) K
Plate, yellow
0.60 × 0.50 × 0.20 mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical *SADABS* (Sheldrick, 1996)
$T_{\min} = 0.958$, $T_{\max} = 0.986$
39404 measured reflections
4454 independent reflections
2900 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.064$
$\theta_{\max} = 30.8°$
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -32 \rightarrow 32$
275 standard reflections
frequency: 1200 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.140$
$S = 1.05$
4454 reflections
204 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 0.1620P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$C8$^{\rm i}$	0.851 (18)	2.523 (18)	3.3375 (17)	160.4 (15)

Symmetry code: (i) $x, 1 + y, z$.

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [U(H) = 1.2 $U_{\rm eq}$(C) or U(H) = 1.5 $U_{\rm eq}$(C$_{\rm methyl}$)] using a riding model with aromatic C—H = 0.95 Å or methyl C—H = 0.98 Å, respectively. The H atom bonded to N was refined isotropically. The methyl group was allowed to rotate about its local threefold axis.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Siemens, 1991).

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