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Redetermination of 9-nitroanthracene at 100 K

Neil M. Glagovich,[a] Paul C. D. Foss,[a] Oliver Michalewski,[a] Elizabeth M. Reed,[a] Katherine E. Strathearn,[a] Yan F. Weiner,[a] Guy Crundwell,[a]* James B. Updegraff III,[b] Matthias Zeller[b] and Allen D. Hunter[b]

[a]Department of Chemistry, Central Connecticut State University, New Britain, CT 06053, USA, and [b]Department of Chemistry, Youngstown State University, One University Plaza, Youngstown, Ohio 44555-3663, USA

Correspondence e-mail:
crundwellg@mail.ccsu.edu

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The title compound, $C_{14}H_9NO_2$, was synthesized by the electrophilic aromatic substitution reaction between anthracene and nitric acid. The crystal structure is a low-temperature (100 K) redetermination of a previously reported room-temperature structure [Trotter (1959). *Acta Cryst.* **12**, 237–242].

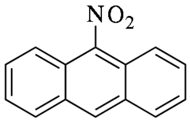
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.148
Data-to-parameter ratio = 16.5

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

Comment

The title compound, (I) (Fig. 1), was synthesized as a precursor to 9-aminoanthracene (to be used in imine formation with a variety of aromatic ketones). Although the crystal structure of 9-nitroanthracene has already been determined (Trotter, 1959), our synthesis resulted in crystals giving a much better R value than previously reported; we have determined the angle between the planes of the nitro group and the anthracene ring system to be 70.23 (4)° instead of 85° as previously reported (Trotter, 1959).



(I)

Experimental

9–Nitroanthracene was synthesized by a modification of a previously reported synthesis (Braun *et al.*, 1960). To a 50 ml Erlenmeyer flask chilled in an ice-water bath were added anthracene (3 g, 17 mmol) and glacial acetic acid (12 ml, 210 mmol). To the chilled suspension was added concentrated nitric acid (1.2 ml, 19 mmol) with vigorous stirring over a 15 min period. After this time, the solution was stirred

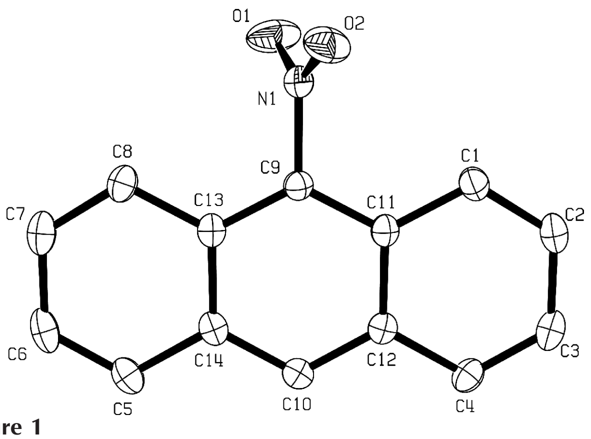


Figure 1
A view of the molecule of (I) (Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

for an additional hour, resulting in a brown–yellow solution. The solution was filtered to remove any unreacted anthracene and the filtrate was added to 50/50 glacial acetic acid/concentrated hydrochloric acid solution (15 ml). The pale-yellow precipitate (9-nitro-10-chloro-9,10-dihydroanthracene) was isolated by vacuum filtration and the filtrate was washed with two 5 ml portions of glacial acetic acid and then with cold water until the washings were neutral to litmus. The resulting yellow solid was triturated with 9 ml of warm (333–343 K) 10% (*w*/*w*) sodium hydroxide solution, giving an orange solid. The crude product was isolated by vacuum filtration, and washed with two 6 ml portions of a 10% (*w*/*w*) sodium hydroxide solution and then with cold water until the washings were neutral to litmus. The resulting orange solid was recrystallized from glacial acetic acid to yield 3.3 g of 9-nitroanthracene (87%) (m.p. 417.5 K). IR ($CHCl_3$, cm^{-1}): 3057, 1518, 1456, 1444, 1374, 1276, 890, 774, 727; ^1H NMR (300 MHz, $CDCl_3$): δ 8.611 (*s*, 1H), 8.068 (*d*, 2H, J = 8.5 Hz), 7.956 (*d*, 2H, J = 8.8 Hz), 7.657 (*t*, 2H, J = 8.5 Hz), 7.565 (*t*, 2H, J = 8.8 Hz); ^{13}C NMR (300 MHz, $CDCl_3$): δ 143.99, 131.07, 130.62, 129.13, 128.64, 126.48, 122.93, 121.67; UV/Vis (CH_2Cl_2; λ_{max}) 217, 252, 348, 366, 384 nm; MS, calculated for $C_{14}H_9NO_2$: M^+: 223, measured: 223.

Crystal data

$C_{14}H_9NO_2$
M_r = 223.22
Monoclinic, $P2_1/c$
a = 10.5117 (7) Å
b = 13.5401 (9) Å
c = 7.4285 (5) Å
β = 103.197 (1)°
V = 1029.37 (12) Å^3
Z = 4
D_x = 1.440 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5299 reflections
θ = 2.5–28.3°
μ = 0.10 mm^{-1}
T = 100 (2) K
Needle, yellow
0.40 × 0.16 × 0.16 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.859, T_{max} = 0.984
10 391 measured reflections
2545 independent reflections
2182 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 28.3°
$h = -14 \rightarrow 14$
$k = -18 \rightarrow 18$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.148
S = 1.28
2545 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0913P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.017
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

H atoms were placed in calculated positions, with C—H distances of 0.95 Å, and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997–1999); cell refinement: *SAINT-Plus* (Bruker, 1997–1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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