1-Ethoxy-4-nitrobenzene

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The title compound, C<sub>8</sub>H<sub>9</sub>NO<sub>3</sub>, was synthesized from 4-nitrophenol and ethyl iodide via the Williamson ether synthesis. The crystal structure has been determined at 100 K.

Comment

1-Ethoxy-4-nitrobenzene, (I) (Fig. 1), also known as p-nitrophenetole, was synthesized by undergraduate chemistry students as part of an undergraduate laboratory course. The students were preparing a variety of ethers in an effort to design a laboratory that could be incorporated into the undergraduate organic chemistry curriculum. Of the ethers prepared, 1-ethoxy-4-nitrobenzene required the shortest reaction time, gave the best yield, and was the least problematic in terms of isolation. In addition, upon standing, a solution of (I) in ethanol produced very large and well defined crystals. Because the crystal structure of this ether has not been previously reported, it was decided that it would be an interesting extension of the laboratory experience to have the students in the class solve the structure from the X-ray diffraction data.

Experimental

To a 100 ml round-bottomed flask equipped with a reflux condenser were added 15 ml of an 8% (w/w) sodium hydroxide solution, p-nitrophenol (2.78 g, 19 mmol), ethyl iodide (3.2 g, 21 mmol) and acetone (25 ml). The resulting mixture was heated under reflux for 90 min. After this time, the resulting yellow solution was poured over 75 g of ice, and stirred until the ice melted. The crude yellow solid was collected by vacuum filtration and recrystallized from 95% ethanol to yield p-nitrophenetole (yield: 2.64 g, 83%; mp. 330.2 K). The NMR data agree with the published literature (Heathcock, 1962). IR (CHCl₃): 3115, 3098, 2987, 1609, 1597, 1497, 1474, 1342, 1329, 1302, 1261, 1176, 1090, 1041, 920, 860, 851, 751, 655 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 8.204 (d, 2H, J = 10.5 Hz), 6.944 (d, 2H, J = 10.5 Hz), 4.130 (q, 2H, J = 7.0 Hz), 1.465 (t, 3H, J = 7.0 Hz); ¹³C NMR...
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(300 MHz, CDCl₃): δ 164.19, 141.39, 125.89, 114.48, 64.53, 14.56; MS (calculated for C₈H₉NO₃): M⁺: 167, measured: 167.

Crystal data

\[ \text{C}_8\text{H}_9\text{NO}_3 \]

Mr = 167.16
Monoclinic, \( P2_1/c \)
\( a = 7.2796 \) Å
\( b = 11.7664 \) Å
\( c = 9.4285 \) Å
\( \beta = 104.957 \)°
\( V = 780.23 \) Å³
\( Z = 4 \)

Data collection

Bruker SMART APEX diffractometer
\( \omega \) scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
\( T_{\text{min}} = 0.948, T_{\text{max}} = 0.969 \)
1894 independent reflections
1894 reflections with \( I > 2\sigma(I) \)
Refinement

Refinement on \( F^2 \)
\( R[F^2 > 2\sigma(F^2)] = 0.048 \)
\( wR(F^2) = 0.129 \)
\( S = 1.22 \)
1936 reflections
136 parameters
All H-atom parameters refined

\[ D_c = 1.423 \text{ Mg m}^{-3} \]
Mo Kα radiation
Cell parameters from 6367 reflections
\( \theta = 2.2–28.3\° \)
\( \mu = 0.11 \text{ mm}^{-1} \)
\( T = 100 (2) \text{K} \)
Block, colorless
0.45 × 0.45 × 0.30 mm

1936 independent reflections
1894 reflections with \( I > 2\sigma(I) \)
\( R_{	ext{int}} = 0.021 \)
\( \theta_{\text{max}} = 28.3\° \)
\( h = -9 \rightarrow 9 \)
\( k = -15 \rightarrow 15 \)
\( l = -12 \rightarrow 12 \)

w = 1/[\sigma^2(F^2) + (0.0575P)^2 + 0.3353P]
where \( P = (F^2 + 2F_C^2)/3 \)
\( \Delta \rho_{\text{max}} = 0.37 \text{e Å}^{-3} \)
\( \Delta \rho_{\text{min}} = -0.23 \text{e Å}^{-3} \)

Positional and isotropic displacement parameters for all H atoms were allowed to refine after their location in a difference map.

Data collection: SMART (Bruker, 1997–1999); cell refinement: SAINT-Plus (Bruker, 1997–1999); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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References