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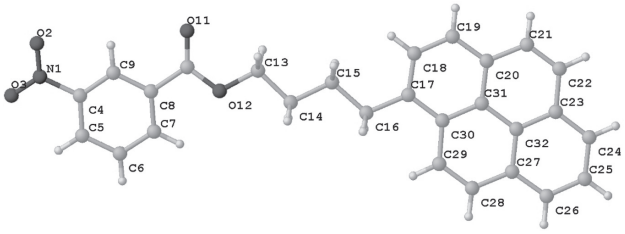
The crystal structure of 4-(pyren-1-yl)butyl-3-nitrobenzoate, $C_{27}H_{21}NO_4$



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Abstract

$C_{27}H_{21}NO_4$, triclinic, $P\bar{1}$ (no. 2), $a = 8.1182(5)$ Å, $b = 9.0097(5)$ Å, $c = 14.8013(10)$ Å, $\alpha = 72.603(2)°$, $\beta = 82.642(2)°$, $\gamma = 79.351(3)°$, $V = 1012.23(11)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0367$, $wR_{ref}(F^2) = 0.1018$, $T = 150$ K.

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The molecular 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.

Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	0.27 × 0.20 × 0.06 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE Bruker AXS, φ and ω-scans
θ_{max}, completeness:	25.0°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12408, 3506, 0.040
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2839
$N(param)_{refined}$:	290
Programs:	Bruker [1], Olex2 [2, 3], SHELX [4]

Source of material

As proposed by Moscoso *et al.* [5], an equimolar quantity of 3,5-dinitrobenzoic chloride and 4-(pyren-1-yl)butanol is added in a flask with dry THF. The reaction was carried out for 24 h at room temperature forming an insoluble yellow precipitate, which was washed with a saturated solution of $NaHCO_3$, and finally washed with hot ethanol. 1.08 g of product was obtained with a 52.4% yield. **^{1}H NMR** (Bruker WM300, 300 MHz, DMSO-d_6, δ (ppm)): 8.63–7.95 (m, 12 H, Ar—H), 7.77 (q, $J = 7.9$ Hz, 1 H, Ar—H); 4.43 (t, 2 H, CH_2—OR), 3.44 (c, 2 H, CH_2-Piren), 2.1–1.8 (m, 4 H, 2xCH_2), **^{13}C-NMR** (DMSO-d_6, δ (ppm)): 164.44; 148.29; 137.06; 135.55; 131.70; 131.34; 131.10; 130.85; 129.75; 128.54; 128.12; 127.98; 127.91; 127.67; 126.96; 126.60; 125.40; 125.24; 124.70; 124.59; 123.90; 65.83; 32.60; 31.17; 28.44. Crystals of 4-(pyren-1-yl)butyl-3-nitrobenzoate were prepared by dissolving the polycrystalline material in boiling chloroform (0.5 ml) and then hot methanol was added dropwise (0.5 ml). The mixture was allowed to crystallize for a week until the appearance of yellow plates.

Experimental details

Using Olex2 [2], the structure was solved, with the olex2.solve [3] using Charge Flipping and refined with the ShelXL [4] refinement package. H atoms were finally included in their calculated positions and treated as riding on their parent atom with constrained thermal parameters, the constraint distances of C—H ranging from 0.95 Å to 1.00 Å. Due to the data collection strategy we only obtained a completeness of 98%.

Comment

Nitro compounds are of interest due to the electrochemical properties they possess. This electrochemistry behavior was studied by many scientist [6–8]. The nitro compounds can be used as a mediator in NADH oxidation process due to oxidation mechanism, *via* two electrons and two protons [9–11]. The nitro compounds mediators can be used as a electrochemistry biosensor, using nanomaterials such as multiwalled carbon nanotubes under physisorption. The inclusion of pyrene rings major interaction with multiwalled

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.83076(16)	1.05762(16)	1.10376(9)	0.0276(3)
O2	0.83378(19)	0.94104(14)	1.17275(8)	0.0487(4)
O3	0.87714(16)	1.17906(15)	1.10301(9)	0.0418(3)
C4	0.76527(17)	1.05205(17)	1.01665(10)	0.0201(3)
C5	0.73526(18)	1.19088(18)	0.94528(10)	0.0239(3)
H5	0.756976	1.287012	0.951903	0.029*
C6	0.6724(2)	1.18593(18)	0.86357(10)	0.0271(4)
H6	0.651412	1.279620	0.813072	0.032*
C7	0.64024(18)	1.04541(18)	0.85522(10)	0.0229(3)
H7	0.597185	1.043299	0.798957	0.028*
C8	0.67034(17)	0.90695(16)	0.92848(9)	0.0182(3)
C9	0.73446(17)	0.90967(17)	1.01052(10)	0.0197(3)
H9	0.756539	0.816183	1.061035	0.024*
C10	0.63480(17)	0.75398(17)	0.92159(10)	0.0197(3)
O11	0.68100(14)	0.62745(12)	0.97618(7)	0.0278(3)
O12	0.54542(12)	0.77620(11)	0.84744(7)	0.0218(3)
C13	0.51779(19)	0.63498(16)	0.82677(10)	0.0214(3)
H13A	0.444579	0.575854	0.878518	0.026*
H13B	0.626225	0.565817	0.821059	0.026*
C14	0.43487(18)	0.68615(16)	0.73441(10)	0.0196(3)
H14A	0.501862	0.756500	0.685047	0.023*
H14B	0.321385	0.745713	0.742829	0.023*
C15	0.42071(17)	0.54349(16)	0.70245(9)	0.0178(3)
H15A	0.356212	0.472486	0.752977	0.021*
H15B	0.534778	0.485177	0.693767	0.021*
C16	0.33510(17)	0.58713(16)	0.61015(10)	0.0175(3)
H16A	0.222174	0.647295	0.619118	0.021*
H16B	0.400598	0.657890	0.560092	0.021*
C17	0.31535(17)	0.45035(16)	0.57506(9)	0.0166(3)
C18	0.36880(18)	0.29499(16)	0.62612(10)	0.0209(3)
H18	0.419498	0.275506	0.683794	0.025*
C19	0.35055(18)	0.16809(17)	0.59569(10)	0.0224(3)
H19	0.387973	0.064064	0.632839	0.027*
C20	0.27789(17)	0.19133(16)	0.51116(10)	0.0190(3)
C21	0.25653(19)	0.06299(17)	0.47681(11)	0.0253(4)
H21	0.292841	−0.042090	0.512807	0.030*
C22	0.18653(19)	0.08849(17)	0.39514(11)	0.0256(4)
H22	0.174814	0.001010	0.374511	0.031*
C23	0.12894(17)	0.24460(17)	0.33820(10)	0.0205(3)
C24	0.05671(18)	0.27348(18)	0.25267(11)	0.0239(3)
H24	0.045613	0.187316	0.230602	0.029*
C25	0.00081(18)	0.42595(19)	0.19930(10)	0.0248(4)
H25	−0.046738	0.443223	0.140927	0.030*
C26	0.01424(17)	0.55305(18)	0.23101(10)	0.0224(3)
H26	−0.025968	0.656794	0.194513	0.027*
C27	0.08643(16)	0.53044(16)	0.31627(9)	0.0177(3)
C28	0.10190(17)	0.65857(16)	0.35174(10)	0.0191(3)
H28	0.059893	0.763155	0.317314	0.023*
C29	0.17526(17)	0.63347(16)	0.43342(10)	0.0174(3)
H29	0.184783	0.721400	0.454194	0.021*
C30	0.23921(16)	0.47778(16)	0.48970(9)	0.0155(3)
C31	0.22156(16)	0.34829(16)	0.45726(9)	0.0163(3)
C32	0.14586(16)	0.37479(16)	0.37053(9)	0.0168(3)

carbon nanotubes as we previously reported [5]. The N—O bond lengths in the nitro group range from 1.226(17) to 1.2182(16) Å. The angle between O3—N1—O2 is 123.91(13)°, O12—C10—O11 is 124.91°, C13—O12—C10 is 116.38(12)°, O2—N1—C4 is 118.36(13)°, O3—N1—C4 is 117.72(13)°, C9—C4—N1 is 118.96(13)° and C5—C4—N1 is 117.99(13)°. These angles and distances are similar to those reported for 1,2-dimethyl-3,4-dinitrobenzene, $C_8H_8N_2O_4$ [12]. In the crystal structure of the title compound, coplanarity of the nitro group with the pyrene group is observed.

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