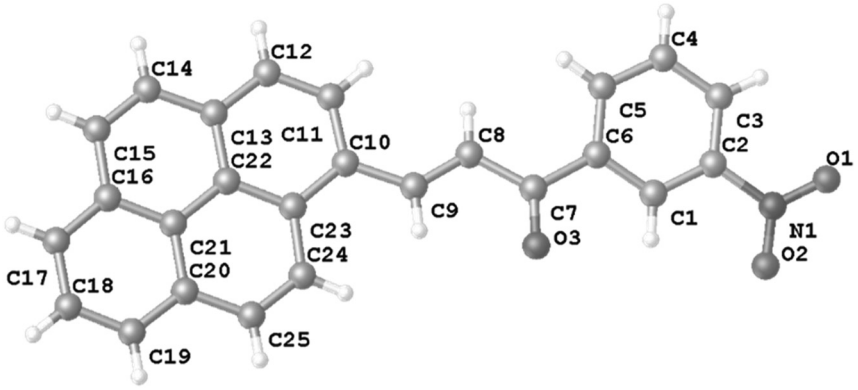
Claudio Barrientos, J. Arturo Squella and Silvana Moris*

The crystal structure of (*E*)-1-((3)-nitrophenyl) pyren-3-(pyren-1-yl)prop-2-en-1-one, $C_{25}H_{15}NO_3$



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Abstract

$C_{25}H_{15}NO_3$, triclinic, $P\bar{1}$ (no. 2), a = 8.4899(3) Å, b = 10.1383(4) Å, c = 11.3806(5) Å, α = 78.373(1)°, β = 72.020(1)°, γ = 73.381(1)°, V = 885.89(6) Å^3, Z = 2, $R_{gt}(F)$ = 0.0439, $wR_{ref}(F^2)$ = 0.1290, T = 296.15 K.

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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:	Orange block
Size:	0.16 × 0.16 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25692, 3611, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2790
$N(param)_{refined}$:	263
Programs:	Olex2,[1,2] SHELX,[3] PLATON[4]

1 Source of materials

Equimolar amounts of 1-pyrencarboxaldehyde and 3-nitroacetophenone were carried out under reflux for 8 h in ethanol with two drops of piperidine as a catalyst.[5] The solid product precipitates in the reaction medium, and then is filtered under vacuum and washed with hot ethanol to eliminate all traces of starting reagents. The product is a yellow solid. To obtain single crystals, the solid product was placed in hot DMF and left to crystallize for two weeks, or until orange blocks started to emerge (*E*)-1-((3)-nitrophenyl)-3-(pyren-1-yl)prop-2-en-1-one. Yield 85 %. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.97 (d, J = 15.3 Hz, 1H), 8.92 (t, J = 2.0 Hz, 1H), 8.89 (d, J = 8.2 Hz, 1H), 8.71 (dt, J = 7.9, 1.3 Hz, 1H), 8.65 (d, J = 9.4 Hz, 1H), 8.53–8.48 (m, 1H),8.42–8.22 (m, 7H), 8.14 (t, J = 7.6 Hz, 1H), 7.91 (t, J = 8.0 Hz, 1H). **^{13}C NMR** (101 MHz, DMSO) δ 187.80, 148.72, 141.52, 139.35, 135.27, 133.27, 131.29, 131.09, 130.63, 130.41, 129.51, 129.42, 128.33, 127.86, 127.82, 127.18, 126.86, 126.62, 125.74, 125.73, 124.55, 124.19, 123.79, 123.39, 122.76.

2 Experimental details

Using Olex2,[1] with the olex2.solve[2] using Charge Flipping and refined with the SHELXL[3] the structure was solved.

***Corresponding author: Silvana Moris**, Centro de Investigación de Estudios Avanzados del Maule (CIEAM), Vicerrectoría de Investigación y Postgrado, Universidad Católica del Maule, Talca, Chile,
E-mail: smoris@ucm.cl. https://orcid.org/0000-0002-0330-8751
Claudio Barrientos, Centro de Investigación de Estudios Avanzados del Maule (CIEAM), Vicerrectoría de Investigación y Postgrado, Universidad Católica del Maule, Talca, Chile
J. Arturo Squella, Facultad de Ciencias Quimicas y Farmaceuticas, Centro de Investigacion de procesos redox (CiPRex), Universidad de Chile, Santiago, Chile

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.82079 (18)	−0.09534 (14)	0.82596 (14)	0.0484 (4)
H1	0.88114 (18)	−0.02805 (14)	0.78625 (14)	0.0581 (4)*
C2	0.90100 (18)	−0.22121 (15)	0.87785 (14)	0.0489 (4)
C3	0.8171 (2)	−0.32418 (15)	0.93693 (14)	0.0530 (4)
H3	0.8743 (2)	−0.40886 (15)	0.97073 (14)	0.0636 (5)*
C4	0.6468 (2)	−0.29854 (15)	0.94458 (14)	0.0556 (4)
H4	0.5876 (2)	−0.36661 (15)	0.98418 (14)	0.0667 (5)*
C5	0.56208 (19)	−0.17244 (15)	0.89399 (14)	0.0505 (4)
H5	0.44639 (19)	−0.15632 (15)	0.90054 (14)	0.0606 (4)*
C6	0.64827 (18)	−0.06983 (14)	0.83358 (13)	0.0456 (3)
C7	0.56828 (19)	0.06617 (14)	0.77011 (14)	0.0502 (4)
C8	0.38248 (19)	0.11314 (15)	0.79359 (14)	0.0524 (4)
H8	0.31296 (19)	0.05646 (15)	0.84480 (14)	0.0629 (4)*
C9	0.31403 (19)	0.23666 (15)	0.74157 (14)	0.0503 (4)
H9	0.39159 (19)	0.28787 (15)	0.69282 (14)	0.0604 (4)*
C10	0.13601 (18)	0.30429 (14)	0.74915 (13)	0.0460 (3)
C11	0.0070 (2)	0.23634 (15)	0.81603 (14)	0.0539 (4)
H11	0.0357 (2)	0.14931 (15)	0.86023 (14)	0.0646 (5)*
C12	−0.16074 (19)	0.29460 (16)	0.81814 (15)	0.0553 (4)
H12	−0.24310 (19)	0.24659 (16)	0.86393 (15)	0.0664 (5)*
C13	−0.21009 (18)	0.42467 (15)	0.75282 (13)	0.0481 (3)
C14	−0.38354 (19)	0.48695 (17)	0.74993 (15)	0.0566 (4)
H14	−0.46712 (19)	0.43890 (17)	0.79223 (15)	0.0679 (5)*
C15	−0.42811 (19)	0.61322 (17)	0.68747 (15)	0.0584 (4)
H15	−0.54193 (19)	0.65075 (17)	0.68812 (15)	0.0700 (5)*
C16	−0.30510 (18)	0.69073 (15)	0.62041 (14)	0.0503 (4)
C17	−0.3478 (2)	0.82252 (18)	0.55483 (16)	0.0617 (4)
H17	−0.4609 (2)	0.86207 (18)	0.55453 (16)	0.0741 (5)*
C18	−0.2259 (2)	0.89449 (17)	0.49098 (16)	0.0643 (5)
H18	−0.2574 (2)	0.98193 (17)	0.44825 (16)	0.0772 (5)*
C19	−0.0572 (2)	0.83832 (16)	0.48963 (15)	0.0562 (4)
H19	0.0242 (2)	0.88800 (16)	0.44571 (15)	0.0674 (5)*
C20	−0.00734 (18)	0.70739 (14)	0.55370 (13)	0.0464 (3)
C21	−0.13190 (17)	0.63218 (14)	0.62037 (12)	0.0434 (3)
C22	−0.08343 (17)	0.49822 (14)	0.68613 (12)	0.0420 (3)
C23	0.09093 (17)	0.43875 (14)	0.68442 (12)	0.0426 (3)
C24	0.21231 (18)	0.51862 (15)	0.61550 (14)	0.0492 (4)
H24	0.32675 (18)	0.48167 (15)	0.61317 (14)	0.0591 (4)*
C25	0.16610 (18)	0.64542 (15)	0.55402 (14)	0.0508 (4)
H25	0.24915 (18)	0.69369 (15)	0.51079 (14)	0.0609 (4)*
N1	1.08375 (17)	−0.24715 (14)	0.86821 (14)	0.0632 (4)
O1	1.16062 (16)	−0.36356 (13)	0.89792 (13)	0.0801 (4)
O2	1.15100 (17)	−0.15056 (15)	0.83108 (19)	0.1084 (6)
O3	0.66127 (14)	0.13690 (11)	0.69929 (13)	0.0770 (4)

SADABS-2016/2 (Bruker, 2016/2) was used for absorption correction. Interactions were calculated using Platon.[4]

H atoms were finally included in their calculated positions and treated as riding on their parent atom with constrained thermal parameters as U_{iso}(H) = 1.2 U_{eq}(C), the constraint distances of C–H was 0.93 Å.

3 Comment

Chalcones are organic compounds found in plants and are related to the flavonoid family, structurally present two aromatic fragments linked through a double bond conjugated to a carbonyl,[6] these compounds are vastly studied and synthesized due to their chemical modifications that allow preparing of multiple derivatives including heterocyclic compounds.[7] Some chalcones show biological properties such as analgesic,[8] anti-inflammatory,[9] antiplatelet,[10] antioxidant[11] for example. By other hand, pyrenes are known for optical uses, and pyrene chalcones are molecules that can be transformed into other structures with interesting photo physicochemical properties.[12] In this structure there are two crystallographically independent molecules in the asymmetric unit. The N–O bond lengths in the nitro group range from 1.2115(18) to 1.2116(17) Å. The angle between O–N–O in these structures is 122.76(15)°, O–N–C range between 118.91(14)° to 118.33(13)°, similar value to reported by us in Barrientos, et al.[13–15] There are no classical hydrogen bonds in the crystal packing,[4] it only exhibits intermolecular C–H···O interactions with distances of 2.376(2)–2.525(2) Å between the donor and acceptor atoms.[16] The crystal structure exhibits N–O···π interactions N(1)–O(1)···Cg(2) = 3.8000(15) Å and N(1)–O(1)···Cg(5) = 3.8326(15) Å.

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