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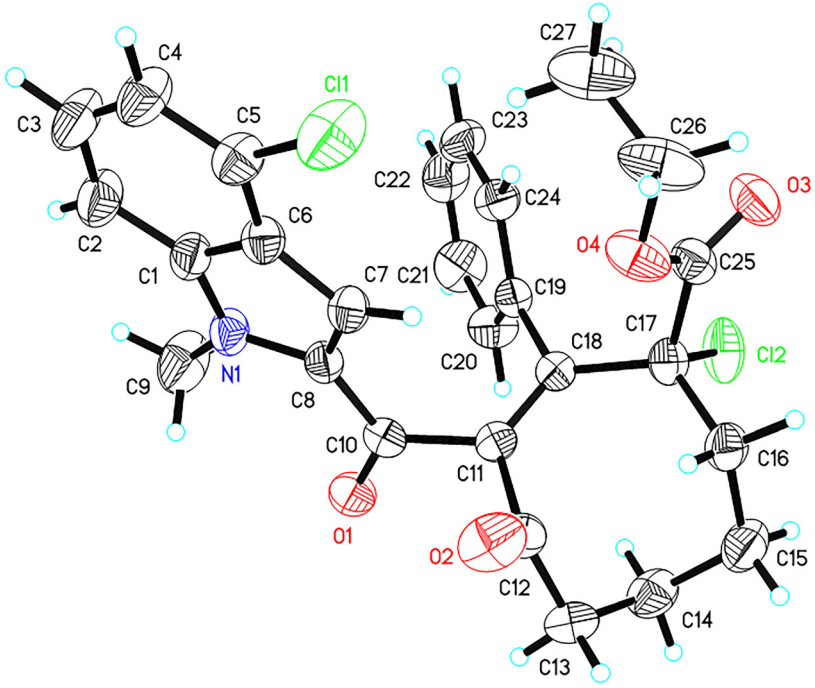
The crystal structure of ethyl (*E*)-1-chloro-3-(4-chloro-1-methyl-1*H*-indole-2-carbonyl)-4-oxo-2-phenylcyclooct-2-ene-1-carboxylate, $C_{27}H_{25}Cl_2NO_4$



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Abstract

$C_{27}H_{25}Cl_2NO_4$, monoclinic, $P2_1/n$ (no. 14), a = 18.9834(7) Å, b = 8.5683(3) Å, c = 30.4308(12) Å, β = 93.832(1)°, V = 4938.7(3) Å^3, Z = 8, $R_{gt}(F)$ = 0.0655, $wR_{ref}(F^2)$ = 0.2089, T = 296(2) K.

CCDC no.: 2306965

Table 1: Data collection and handling.

Crystal:	Colourless prismatic
Size:	0.18 × 0.15 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.30 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	26,914, 9655, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5920
$N(param)_{refined}$:	637
Programs:	Olex2 [1], SHELX [2, 3]

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of material

In a Schlenk tube 1-(4-chloro-1-methyl-1*H*-indol-2-yl)-3-phenyl prop-2-yn-1-one (0.20 mmol, 58.8 mg), ethyl 2-oxocyclohexane-

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.13983 (5)	0.99910 (16)	0.66821 (4)	0.1007 (4)
Cl2	0.56328 (5)	1.03236 (14)	0.74281 (3)	0.0862 (4)
Cl3	0.72987 (7)	0.61476 (18)	0.42226 (4)	0.1129 (5)
Cl4	0.74326 (7)	0.67893 (17)	0.68892 (4)	0.1208 (5)
N1	0.32289 (14)	0.8331 (3)	0.56943 (8)	0.0608 (7)
N2	0.92637 (13)	0.3500 (3)	0.50700 (9)	0.0600 (7)
O1	0.44818 (13)	0.6642 (3)	0.60650 (8)	0.0720 (7)
O2	0.36179 (14)	0.5753 (3)	0.70834 (10)	0.0947 (9)
O3	0.44180 (16)	1.1918 (3)	0.77244 (9)	0.0894 (8)
O4	0.35948 (13)	1.0254 (3)	0.74623 (10)	0.0832 (8)
O5	0.92298 (12)	0.3065 (3)	0.60251 (8)	0.0725 (7)
O6	0.73306 (14)	0.1929 (3)	0.56971 (9)	0.0842 (8)
O7	0.66221 (18)	0.8192 (4)	0.61804 (17)	0.1497 (17)
O8	0.66906 (17)	0.6289 (5)	0.56847 (13)	0.1171 (11)
C1	0.25735 (17)	0.9024 (4)	0.56700 (11)	0.0608 (8)
C2	0.2159 (2)	0.9537 (5)	0.53050 (12)	0.0841 (12)
H2	0.230486	0.942438	0.502110	0.101*
C3	0.1525 (2)	1.0218 (6)	0.53812 (14)	0.0934 (14)
H3	0.124111	1.058612	0.514255	0.112*
C4	0.12911 (19)	1.0378 (5)	0.58018 (13)	0.0825 (11)
H4	0.085755	1.084524	0.584055	0.099*
C5	0.16962 (17)	0.9850 (4)	0.61578 (12)	0.0651 (9)
C6	0.23608 (16)	0.9184 (4)	0.61025 (10)	0.0557 (8)
C7	0.29169 (15)	0.8620 (4)	0.63871 (10)	0.0538 (7)
H7	0.292684	0.859388	0.669302	0.065*
C8	0.34404 (15)	0.8116 (3)	0.61341 (10)	0.0513 (7)
C9	0.3637 (2)	0.8033 (7)	0.53197 (13)	0.1068 (16)
H9A	0.405133	0.867981	0.533790	0.160*
H9B	0.335742	0.826643	0.505344	0.160*
H9C	0.377520	0.695523	0.531879	0.160*
C10	0.41089 (15)	0.7415 (3)	0.62926 (10)	0.0522 (7)
C11	0.43230 (13)	0.7657 (3)	0.67776 (9)	0.0468 (7)
C12	0.42121 (17)	0.6187 (4)	0.70393 (11)	0.0597 (8)
C13	0.4844 (2)	0.5237 (4)	0.71808 (14)	0.0764 (10)
H13A	0.498240	0.464358	0.692915	0.092*
H13B	0.470970	0.449515	0.740082	0.092*
C14	0.54815 (18)	0.6132 (5)	0.73685 (13)	0.0774 (10)
H14A	0.562513	0.686926	0.714985	0.093*
H14B	0.586844	0.540789	0.743026	0.093*
C15	0.5343 (2)	0.7007 (5)	0.77864 (13)	0.0843 (11)
H15A	0.577277	0.754804	0.788745	0.101*
H15B	0.524133	0.625082	0.801065	0.101*
C16	0.47443 (19)	0.8189 (4)	0.77495 (10)	0.0647 (9)
H16A	0.472840	0.870263	0.803274	0.078*
H16B	0.430577	0.761609	0.769871	0.078*
C17	0.47587 (16)	0.9466 (4)	0.73949 (10)	0.0557 (8)
C18	0.45719 (14)	0.9030 (3)	0.69149 (9)	0.0458 (6)
C19	0.46894 (15)	1.0286 (3)	0.65842 (9)	0.0499 (7)
C20	0.52654 (19)	1.0166 (4)	0.63301 (12)	0.0709 (10)
H20	0.556096	0.930222	0.636244	0.085*
C21	0.5407 (2)	1.1311 (5)	0.60295 (14)	0.0893 (12)
H21	0.579609	1.121406	0.586167	0.107*
C22	0.4979 (3)	1.2581 (5)	0.59776 (13)	0.0889 (13)
H22	0.507725	1.335801	0.577746	0.107*
C23	0.4401 (2)	1.2705 (4)	0.62228 (14)	0.0799 (11)
H23	0.410327	1.356377	0.618364	0.096*
C24	0.42535 (18)	1.1577 (4)	0.65265 (11)	0.0636 (8)
H24	0.386219	1.168181	0.669234	0.076*
C25	0.42448 (19)	1.0730 (4)	0.75449 (11)	0.0617 (8)
C26	0.3028 (3)	1.1203 (6)	0.7631 (2)	0.1170 (18)
H26A	0.264467	1.052851	0.770732	0.140*
H26B	0.320554	1.174335	0.789576	0.140*
C27	0.2767 (3)	1.2327 (6)	0.7308 (2)	0.1231 (19)
H27A	0.312314	1.310109	0.726903	0.185*
H27B	0.235211	1.282072	0.740672	0.185*
H27C	0.265205	1.180804	0.703292	0.185*
C28	0.90705 (17)	0.3950 (4)	0.46461 (11)	0.0615 (8)
C29	0.9439 (2)	0.3793 (5)	0.42650 (13)	0.0875 (12)
H29	0.987704	0.330515	0.427086	0.105*
C30	0.9127 (3)	0.4388 (6)	0.38857 (14)	0.1040 (15)
H30	0.936015	0.430085	0.362777	0.125*
C31	0.8480 (3)	0.5110 (6)	0.38690 (13)	0.0991 (14)
H31	0.828870	0.550938	0.360292	0.119*
C32	0.8111 (2)	0.5252 (5)	0.42406 (12)	0.0770 (10)
C33	0.84058 (18)	0.4667 (4)	0.46427 (10)	0.0605 (8)
C34	0.81928 (16)	0.4601 (4)	0.50777 (10)	0.0578 (8)
H34	0.776847	0.497114	0.517306	0.069*
C35	0.87177 (15)	0.3901 (4)	0.53334 (10)	0.0537 (7)
C36	0.99038 (18)	0.2619 (5)	0.51948 (13)	0.0790 (11)
H36A	0.980129	0.152234	0.518264	0.119*
H36B	1.007193	0.289777	0.548894	0.119*
H36C	1.025931	0.285792	0.499509	0.119*
C37	0.87321 (16)	0.3605 (4)	0.58052 (10)	0.0521 (7)
C38	0.80456 (15)	0.3918 (4)	0.60193 (9)	0.0515 (7)
C39	0.76223 (18)	0.2432 (4)	0.60341 (11)	0.0627 (8)
C40	0.7642 (2)	0.1554 (5)	0.64586 (12)	0.0813 (11)
H40A	0.808187	0.097665	0.649131	0.098*
H40B	0.726008	0.079914	0.644256	0.098*
C41	0.7579 (2)	0.2554 (5)	0.68672 (12)	0.0902 (12)
H41A	0.764025	0.189543	0.712622	0.108*
H41B	0.795560	0.332083	0.688323	0.108*
C42	0.6882 (3)	0.3385 (6)	0.68722 (16)	0.1103 (16)
H42A	0.688501	0.401022	0.713799	0.132*
H42B	0.651337	0.260690	0.688950	0.132*
C43	0.6684 (2)	0.4459 (5)	0.64730 (16)	0.0922 (13)
H43A	0.662194	0.380936	0.621221	0.111*
H43B	0.623200	0.493808	0.651800	0.111*
C44	0.72069 (18)	0.5754 (5)	0.63799 (12)	0.0708 (10)
C45	0.78974 (15)	0.5326 (4)	0.61715 (10)	0.0541 (7)
C46	0.84065 (15)	0.6652 (4)	0.61438 (10)	0.0525 (7)
C47	0.89713 (17)	0.6772 (4)	0.64509 (10)	0.0609 (8)
H47	0.904913	0.598641	0.665950	0.073*
C48	0.94255 (18)	0.8038 (4)	0.64549 (12)	0.0695 (9)
H48	0.980329	0.809723	0.666519	0.083*
C49	0.93187 (18)	0.9195 (4)	0.61510 (11)	0.0645 (9)
H49	0.961561	1.005952	0.615876	0.077*
C50	0.87774 (19)	0.9087 (4)	0.58359 (11)	0.0653 (9)
H50	0.871446	0.986461	0.562379	0.078*
C51	0.83196 (17)	0.7827 (4)	0.58285 (11)	0.0608 (8)
H51	0.795154	0.776564	0.561162	0.073*
C52	0.6812 (2)	0.6927 (6)	0.6078 (2)	0.0969 (14)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C53[a]	0.6371 (6)	0.728 (2)	0.5324 (4)	0.172 (4)
H53A[a]	0.637204	0.836286	0.541588	0.206*
H53B[a]	0.664249	0.719288	0.506631	0.206*
C54[a]	0.5664 (5)	0.6772 (17)	0.5220 (4)	0.176 (4)
H54A[a]	0.566955	0.574121	0.509652	0.265*
H54B[a]	0.543614	0.747510	0.500972	0.265*
H54C[a]	0.541156	0.675927	0.548254	0.265*
C53′[b]	0.6058 (8)	0.691 (3)	0.5483 (6)	0.177 (4)
H53C[b]	0.581798	0.758026	0.568152	0.212*
H53D[b]	0.573887	0.609251	0.537247	0.212*
C54′[b]	0.6342 (10)	0.776 (3)	0.5136 (6)	0.179 (4)
H54D[b]	0.671929	0.841770	0.525421	0.268*
H54E[b]	0.597881	0.840121	0.499325	0.268*
H54F[b]	0.651931	0.705256	0.492627	0.268*

[a]Occupancy: 0.6, [b]Occupancy: 0.4.

1-carboxylate (0.40 mmol, 68.1 mg, 63 μL), Cs_2CO_3 (0.4 mmol, 130.3 mg) and DMSO (2.0 mL) were stirred at 60 °C under N_2. After 2 h, $CuCl_2$ (26.9 mg, 0.2 mmol) and $K_2S_2O_8$ (162.2 mg, 0.6 mmol) were added. After the completion of the addition, the reaction mixture was allowed to react at 100 °C for 2 h in air. The reaction mixture was cooled to room temperature and was treated with H_2O, then extracted with EA and dried over anhydrous Na_2SO_4. After removal of the EA, the residue was purified by chromatography on basic silica gel (PE:EA = 5:1) to afford desired compound (yellow solid, 66.5 mg, 67 %). Single crystals were obtained by crystallization of the title compound from a mixture of dichloromethane (10 mL) and petroleum ether (2 mL).

2 Experimental details

Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program and refined with the SHELXL [3] refinement package.

3 Comment

Cyclooctanone and indole scaffold are widely present in many natural products and drug molecules [4–7]. In this crystal structure, there is an indole ring, a cyclooctanone ring and a benzene ring. The indole ring in the structure is substituted with a chlorine atom at the 4-position. The bond length of Cl(1)–C(5) is 1.732 Å, which is similar to that reported [8]. There is also a Cl bond on the ring of cyclooctanone, and the bond length of Cl(2)–C(17) is 1.811, which is a typical. There are two ketone carbonyls and one ester carbonyl in this structure. The bond lengths of the three carbonyl groups are almost the same (1.219 Å, 1.203 Å and 1.191 Å) [9, 10].

The complete set of X-ray diffraction data for the title compound was deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 2306965).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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