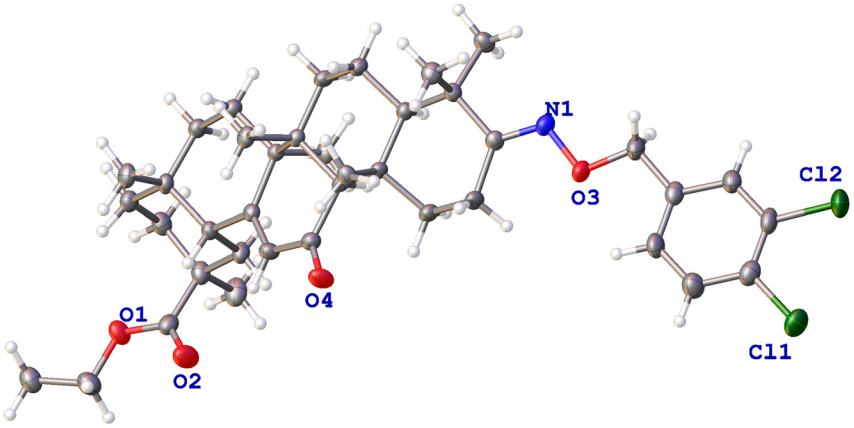
Zhang-Chao Dong, Hui Shen, Ze-Bo Mu* and Kai-Hua Xu

Synthesis and crystal structure of ethyl (2*S*,4a*S*,6a*S*,6b*R*,8a*R*,12a*S*,12b*R*,14b*R*,*E*)-10-(((3,4-dichlorobenzyl)oxy)imino)-2,4a,6a,6b,9,9,12a-heptamethyl-13-oxo-1,2,3,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,12b,13,14b-icosahydropicene-2-carboxylate



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Abstract

$C_{39}H_{53}Cl_2NO_4$, monoclinic, $P2_1$ (no. 4), a = 7.55730(10) Å, b = 11.0989(2) Å, c = 20.9222(3) Å, β = 91.426(1)°, V = 1754.36(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0360, $wR_{ref}(F^2)$ = 0.1028, T = 169.98(10) 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:	Colourless block
Size:	0.14 × 0.12 × 0.10 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	1.99 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC12 (RINC), ω
θ_{max}, completeness:	71.6°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8505, 4877, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4,771
$N(param)_{refined}$:	423
Programs:	CrysAlis^{PRO},[1] Olex2,[2] SHELX[3]

1 Source of material

An amount of 1 mmol of glycyrrhetinic acid was dissolved in THF. Then, 4.8 mmol of Jones reagent was added and reacted at 0 °C. After that, column chromatography purification was carried out. The obtained compound was dissolved in DMF, and potassium iodide, potassium carbonate and bromoethane were added. The mixture was stirred at room temperature for 6 h. The obtained compound was purified by column chromatography again. The obtained clean

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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}
Cl1	1.22096 (10)	0.45274 (9)	0.02981 (4)	0.0476 (2)
Cl2	0.92423 (11)	0.27093 (10)	−0.01693 (4)	0.0540 (3)
O1	0.5716 (3)	0.5090 (2)	0.91306 (8)	0.0337 (5)
O2	0.7056 (3)	0.5477 (3)	0.82164 (10)	0.0473 (6)
O3	0.5548 (2)	0.4603 (2)	0.23300 (8)	0.0326 (5)
O43	0.5947 (3)	0.6898 (2)	0.54171 (9)	0.0352 (5)
N1	0.3814 (3)	0.4549 (2)	0.25845 (10)	0.0287 (5)
C1	0.1911 (3)	0.4970 (3)	0.83571 (12)	0.0270 (6)
H1A	0.246862	0.565829	0.858566	0.032*
H1B	0.071309	0.486097	0.852884	0.032*
C2	0.2996 (4)	0.3838 (3)	0.85056 (12)	0.0278 (6)
H2A	0.236129	0.312554	0.833108	0.033*
H2B	0.311565	0.373812	0.897487	0.033*
C3	0.4843 (4)	0.3900 (3)	0.82202 (12)	0.0278 (6)
C4	0.4626 (3)	0.4106 (3)	0.74942 (12)	0.0261 (6)
H4A	0.402696	0.339809	0.729909	0.031*
H4B	0.581218	0.417272	0.730731	0.031*
C5	0.3549 (3)	0.5250 (2)	0.73244 (11)	0.0215 (5)
H5	0.424357	0.594957	0.749783	0.026*
C6	0.3420 (3)	0.5418 (2)	0.66026 (11)	0.0204 (5)
C7	0.4691 (3)	0.6044 (3)	0.63163 (12)	0.0259 (5)
H7	0.559667	0.638690	0.658165	0.031*
C8	0.4795 (3)	0.6241 (3)	0.56241 (12)	0.0246 (5)
C9	0.3438 (3)	0.5567 (2)	0.52042 (11)	0.0192 (5)
H9	0.385193	0.471156	0.521125	0.023*
C10	0.1632 (3)	0.5541 (2)	0.55581 (11)	0.0184 (5)
C11	0.1925 (3)	0.4820 (2)	0.62060 (11)	0.0186 (5)
C12	0.0192 (3)	0.4738 (2)	0.65835 (11)	0.0226 (5)
H12A	−0.042582	0.552393	0.655410	0.027*
H12B	−0.058785	0.412680	0.637794	0.027*
C13	0.0462 (3)	0.4411 (3)	0.72908 (11)	0.0228 (5)
H13A	0.095249	0.358533	0.732407	0.027*
H13B	−0.069798	0.441469	0.750035	0.027*
C14	0.1722 (3)	0.5293 (2)	0.76438 (11)	0.0226 (5)
C15	0.0985 (4)	0.6574 (3)	0.75963 (13)	0.0294 (6)
H15A	0.171299	0.711416	0.786357	0.044*
H15B	−0.023570	0.658249	0.774421	0.044*
H15C	0.100347	0.684479	0.715079	0.044*
C16	0.0196 (3)	0.4917 (3)	0.51401 (11)	0.0224 (5)
H16A	0.039965	0.403638	0.515191	0.027*
H16B	−0.097453	0.507371	0.532580	0.027*
C17	0.0150 (3)	0.5332 (3)	0.44461 (11)	0.0229 (5)
H17A	−0.015482	0.619889	0.442541	0.027*
H17B	−0.077167	0.487882	0.420289	0.027*
C18	0.1954 (3)	0.5127 (2)	0.41452 (11)	0.0191 (5)
H18	0.228143	0.428016	0.426108	0.023*
C19	0.3394 (3)	0.5920 (2)	0.44791 (11)	0.0207 (5)
C20	0.3104 (4)	0.7280 (3)	0.43733 (12)	0.0251 (5)
H20A	0.183825	0.746359	0.439429	0.038*
H20B	0.353531	0.750794	0.395240	0.038*
H20C	0.375317	0.773218	0.470584	0.038*
C21	0.5187 (3)	0.5533 (3)	0.41996 (12)	0.0256 (6)
H21A	0.614015	0.603166	0.439659	0.031*
H21B	0.542216	0.468362	0.431825	0.031*
C22	0.5257 (3)	0.5654 (3)	0.34741 (12)	0.0286 (6)
H22A	0.527805	0.651960	0.336123	0.034*
H22B	0.637056	0.528850	0.332728	0.034*
C23	0.3725 (3)	0.5066 (2)	0.31268 (11)	0.0221 (5)
C24	0.1866 (3)	0.5143 (2)	0.33965 (11)	0.0212 (5)
C25	0.0989 (4)	0.6285 (3)	0.31102 (12)	0.0270 (6)
H25A	0.172689	0.698854	0.321167	0.040*
H25B	−0.018323	0.639304	0.329214	0.040*
H25C	0.086542	0.619766	0.264511	0.040*
C26	0.0785 (4)	0.4052 (3)	0.31627 (13)	0.0283 (6)
H26A	0.079646	0.401576	0.269484	0.042*
H26B	−0.043792	0.413118	0.330233	0.042*
H26C	0.130538	0.331333	0.334164	0.042*
C27	0.5463 (4)	0.3951 (3)	0.17481 (13)	0.0360 (7)
H27A	0.446940	0.425252	0.147555	0.043*
H27B	0.525544	0.308616	0.183596	0.043*
C28	0.7181 (4)	0.4104 (3)	0.14076 (12)	0.0291 (6)
C29	0.8468 (4)	0.4922 (3)	0.15981 (12)	0.0336 (7)
H29	0.829059	0.540555	0.196553	0.040*
C30	1.0028 (4)	0.5049 (3)	0.12586 (14)	0.0391 (7)
H30	1.091000	0.560662	0.139838	0.047*
C31	1.0279 (4)	0.4360 (3)	0.07190 (13)	0.0341 (6)
C32	0.8986 (4)	0.3559 (3)	0.05178 (13)	0.0339 (7)
C33	0.7457 (4)	0.3416 (3)	0.08639 (14)	0.0328 (6)
H33	0.659166	0.284371	0.072806	0.039*
C34	0.5866 (4)	0.2714 (3)	0.83429 (14)	0.0384 (7)
H34A	0.704425	0.277394	0.816008	0.058*
H34B	0.521803	0.204337	0.814222	0.058*
H34C	0.598526	0.257302	0.880438	0.058*
C35	0.5976 (3)	0.4912 (3)	0.85078 (12)	0.0284 (6)
C36	0.6850 (4)	0.5996 (3)	0.94329 (15)	0.0381 (7)
H36A	0.685753	0.673708	0.916977	0.046*
H36B	0.807818	0.569099	0.947610	0.046*
C37	0.6133 (4)	0.6264 (3)	1.00800 (14)	0.0368 (7)
H37A	0.494102	0.660422	1.003088	0.055*
H37B	0.690813	0.684488	1.030198	0.055*
H37C	0.608298	0.551855	1.032983	0.055*
C38	0.0998 (3)	0.6839 (2)	0.56950 (12)	0.0243 (5)
H38A	−0.015326	0.681079	0.589987	0.036*
H38B	0.088556	0.728677	0.529228	0.036*
H38C	0.186057	0.724252	0.597969	0.036*
C44	0.2531 (3)	0.3506 (2)	0.60832 (12)	0.0240 (5)
H44A	0.376569	0.350597	0.595047	0.036*
H44B	0.178098	0.314700	0.574506	0.036*
H44C	0.242953	0.303395	0.647658	0.036*

compound was dissolved in ethanol, hydroxylamine hydrochloride and sodium bicarbonate were added, and refluxed at 90 °C for 14 h. After purification, the obtained compound was dissolved in THF. NaH was added under ice-water bath and stirred for 3 h. Then, 3,4-dichlorobenzyl bromide was added and stirred at room temperature for 15 h. Finally, methanol was added for inactivation, and then purification was carried out to obtain the compound.

2 Comment

Glycyrrhetinic acid is an effective component extracted from the traditional medicinal and edible plant licorice. Compounds obtained by further structural modification of glycyrrhetinic acid have multiple activities and are the focus of people's research recently.[4–6] Glycyrrhetinic acid has a variety of pharmacological activities, such as anti-inflammatory, anti-tumor, and anti-free radical.[7]

The target compound is composed of five six-membered rings connected to a benzene ring through an oxygen atom and a nitrogen atom. There are two chlorine atoms connected to the phenyl ring. The bond lengths and bond angles are within the normal range of the title structure and are consistent with the previous reported range. The first keto group was confirmed by the distance of 1.223(3) Å (C8–O43). The second keto group was confirmed by the distance of 1.206(4) Å (C35–O2), and the carbon and nitrogen are identified by a distance of 1.428(3) Å (N3–O1), respectively.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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

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