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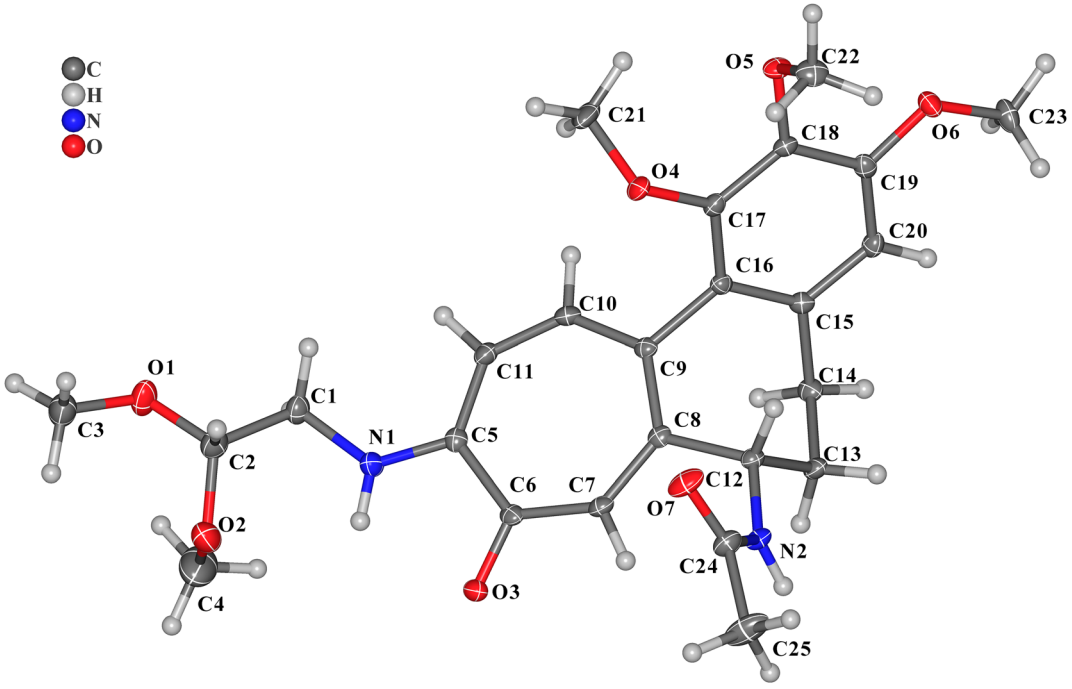
Crystal structure of (*S*)-*N*-(10-((2,2-dimethoxyethyl) amino)-1,2,3-trimethoxy-9-oxo-5,6,7,9-tetrahydrobenzo[*a*]heptalen-7-yl)acetamide, $C_{25}H_{32}N_2O_7$



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Abstract

$C_{25}H_{32}N_2O_7$, Tetragonal, $P4_32_12$, a = 9.0226 (8) Å, c = 59.048 (6) Å, V = 4806.9(10) Å^3, Z = 8, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.112, T = 100 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size	0.12 × 0.11 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker PHOTON-II area detector, φ and ω scans
θ_{max}, completeness:	26.8°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	127638, 5134, 0.058
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4,822
$N(param)_{refined}$:	313
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.1362 (2)	1.0533 (2)	0.32888 (4)	0.0272 (5)
O2	0.9917 (3)	1.0767 (3)	0.29590 (4)	0.0310 (5)
O3	0.7118 (2)	0.7517 (2)	0.26921 (3)	0.0174 (4)
O4	0.5687 (2)	0.3271 (2)	0.36141 (3)	0.0161 (4)
O5	0.4949 (2)	0.0552 (2)	0.38037 (3)	0.0156 (4)
O6	0.4719 (2)	−0.1867 (2)	0.35447 (3)	0.0169 (4)
O7	0.2391 (2)	0.5363 (2)	0.30355 (3)	0.0236 (5)
N1	0.8979 (3)	0.7897 (3)	0.30090 (4)	0.0166 (5)
H1	0.873993	0.848915	0.289643	0.020*
N2	0.3277 (2)	0.3967 (2)	0.27457 (4)	0.0138 (4)
H2	0.323223	0.378569	0.259943	0.017*
C1	1.0151 (3)	0.8412 (3)	0.31577 (5)	0.0185 (6)
H1A	1.112702	0.810875	0.309666	0.022*
H1B	1.003254	0.796577	0.330978	0.022*
C2	1.0074 (3)	1.0091 (3)	0.31747 (5)	0.0223 (6)
H2A	0.919666	1.036293	0.326952	0.027*
C3	1.1330 (4)	1.2042 (3)	0.33578 (6)	0.0277 (7)
H3A	1.108166	1.266975	0.322784	0.042*
H3B	1.230545	1.232512	0.341682	0.042*
H3C	1.058212	1.217167	0.347650	0.042*
C4	1.1124 (5)	1.0517 (5)	0.28124 (7)	0.0484 (11)
H4A	1.113393	1.127753	0.269397	0.073*
H4B	1.102674	0.953604	0.274270	0.073*
H4C	1.205078	1.056321	0.289870	0.073*
C5	0.8217 (3)	0.6625 (3)	0.30230 (4)	0.0133 (5)
C6	0.7092 (3)	0.6512 (3)	0.28423 (4)	0.0131 (5)
C7	0.5996 (3)	0.5382 (3)	0.28323 (4)	0.0140 (5)
H7	0.532036	0.551598	0.271055	0.017*
C8	0.5690 (3)	0.4135 (3)	0.29594 (4)	0.0121 (5)
C9	0.6502 (3)	0.3575 (3)	0.31459 (4)	0.0119 (5)
C10	0.7775 (3)	0.4231 (3)	0.32340 (4)	0.0141 (5)
H10	0.822123	0.367530	0.335239	0.017*
C11	0.8522 (3)	0.5535 (3)	0.31836 (4)	0.0144 (5)
H11	0.938000	0.571241	0.327289	0.017*
C12	0.4371 (3)	0.3187 (3)	0.28818 (4)	0.0128 (5)
H12	0.386439	0.278587	0.301938	0.015*
C13	0.4955 (3)	0.1868 (3)	0.27416 (4)	0.0151 (5)
H13A	0.412577	0.117987	0.271022	0.018*
H13B	0.532924	0.223950	0.259457	0.018*
C14	0.6201 (3)	0.1016 (3)	0.28621 (4)	0.0152 (5)
H14A	0.629548	0.001729	0.279416	0.018*
H14B	0.715177	0.154364	0.283945	0.018*
C15	0.5898 (3)	0.0866 (3)	0.31140 (4)	0.0136 (5)
C16	0.6026 (3)	0.2135 (3)	0.32494 (4)	0.0122 (5)
C17	0.5689 (3)	0.2012 (3)	0.34815 (4)	0.0130 (5)
C18	0.5247 (3)	0.0662 (3)	0.35748 (4)	0.0128 (5)
C19	0.5140 (3)	−0.0596 (3)	0.34367 (4)	0.0143 (5)
C20	0.5460 (3)	−0.0489 (3)	0.32067 (4)	0.0147 (5)
H20	0.538051	−0.133946	0.311261	0.018*
C21	0.6830 (3)	0.3319 (4)	0.37843 (5)	0.0232 (6)
H21A	0.780426	0.331262	0.371085	0.035*
H21B	0.674076	0.245227	0.388337	0.035*
H21C	0.672347	0.422491	0.387440	0.035*
C22	0.3394 (3)	0.0645 (3)	0.38558 (5)	0.0204 (6)
H22A	0.284658	−0.006598	0.376222	0.031*
H22B	0.303792	0.164988	0.382433	0.031*
H22C	0.323926	0.041328	0.401617	0.031*
C23	0.4796 (3)	−0.3212 (3)	0.34170 (5)	0.0200 (6)
H23A	0.404895	−0.319228	0.329664	0.030*
H23B	0.460887	−0.405680	0.351728	0.030*
H23C	0.578449	−0.330791	0.334968	0.030*
C24	0.2328 (3)	0.4959 (3)	0.28364 (5)	0.0179 (6)
C25	0.1182 (4)	0.5555 (4)	0.26727 (5)	0.0322 (8)
H25A	0.020261	0.516551	0.271296	0.048*
H25B	0.143453	0.524597	0.251836	0.048*
H25C	0.116574	0.664000	0.268082	0.048*

1 Source of materials

A solution of colchicine (400 mg, 1.0 mmol) and 2,2-dimethoxyethan-1-amine (1.05 g, 10.0 mmol) was stirred in ethanol at reflux for 3 h. Following evaporation, the mixture was extracted with ethyl acetate (3 × 10 mL), and then the solvent was removed to obtain a yellow solid, which was subsequently dissolved in 90 % methanol and slowly evaporated to obtain crystals.

2 Experimental details

A suitable crystal was selected and tested on a Bruker D8 VENTURE diffractometer[1]. Using Olex2[1], the structure was solved with the SHELXT[2] structure solution program Intrinsic Phasing and refined with the SHELX[3] refinement package using Least Squares minimisation.

3 Comment

Colchicine, one of the oldest drugs and one of the most famous natural molecules, is found mainly in plants of the genus Colchicine, the genus androcymbium, and the genus gloriosa.[4] Colchicine and its derivatives have anti-tumor, anti-gout and other biological activities.[5] We synthesized a colchicine derivative in this study.

A view on the structure of the title molecule is shown in the figure. The compound contains a three-methoxy modified benzene ring and a tropone. And the amine group of 2,2-dimethoxyethan-1-amine successfully replaced the methoxy ring of tropone. N–C single bond distance (N1–C5) is 1.340 (3) Å. N–C single bond distance (N1–C1) is 1.451(3) Å. The dihedral angle of benzene ring and the tropone is 55.635(75)°. All the bond lengths and angles are in the expected ranges.[6,7]

Conflict of interest: The authors declare no conflicts of interest regarding this article.
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Author contribution: All the authors have accepted responsibility for the manuscript and approved submission.

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