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The crystal structure of 3-cyclohexyl-1,5-dioxaspiro[5.5]undecane-2,4-dione, C₁₅H₂₂O₄

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.16 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Rigaku Spider Rapid IP, ω
$\theta_{\max}$, completeness:	27.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13484, 3230, 0.045
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2211
$N(\text{param})_{\text{refined}}$:	172
Programs:	Rigaku [1], SHELX [2, 3]

Abstract

C₁₅H₂₂O₄, monoclinic, $P2_1/c$ (no. 14), $a = 12.7370(5)$ Å, $b = 11.4565(7)$ Å, $c = 10.0430(6)$ Å, $\beta = 103.340(1)^\circ$, $V = 1425.95(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0508$, $wR_{\text{ref}}(F^2) = 0.1406$, $T = 296(2)$ 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.

Source of material

A solution of TEAF was prepared by dropwise adding triethylamine (20 mL) to methanoic acid (5 mL). A 50 mL round-bottomed flask was charged with cyclohexanone (0.49 g, 5 mmol), 1,5-dioxaspiro[5.5]undecane-2,4-dione (0.92 g, 5 mmol) and TEAF (5 mL). The mixture was reacted at room temperature for 48 h, then poured into 100 mL of ice-water. The mixture was filtered and the precipitate was washed three times and dried (M.p. 401.9–402.7 K). The colorless block-shaped crystals of 3-cyclohexyl-1,5-dioxaspiro[5.5]undecane-2,4-dione were obtained from slow evaporation petroleum ether and acetone (2:1 = v/v).

Experimental details

All H atoms were positioned geometrically with $U_{\text{iso}}(\text{H})$ values set at 1.2 times $U_{\text{eq}}(\text{C})$ atoms. The coordinates of the hydrogen atoms of C(8), C(10) and CH₂ group in the cyclohexane ring were included with the C–H distance restrained to 0.98 Å (AFIX 13 option of the SHELXL program [2, 3]), 0.97 Å (AFIX 23 option of the SHELXL program [2, 3]), respectively.

Comment

Recently, a great deal of oxaspirocyclic compounds were designed and synthesized because of their wide application in medicine [4, 5], asymmetric catalysis [6], fluorescent probe [7], fluorescent switch [8], high-density jet fuels [9] and so on. During ten years, our group prepared many oxaspirocyclic compounds [10–12]. In continuation of our work [13], a new compound (3-cyclohexyl-1,5-dioxaspiro[5.5]undecane-2,4-dione) was obtained.

The figure shows the title molecular structure, which includes two cyclohexane rings and one 1,3-dioxane ring. One cyclohexane ring and the 1,3-dioxane ring share the C1 atom and form the 1,5-dioxaspiro[5.5]undecane-2,4-dione group. The other cyclohexane ring and 1,5-dioxaspiro[5.5]undecane-2,4-dione group is linked by C8–C10 bond length of 1.5399(16) Å, which is a typical C–C single bond. Both cyclohexane rings both adopt a chair-like configuration, with puckering parameters for (ring C1–C6): $Q = 0.5615$ Å, $q_2 = 0.026$ Å, $q_3 = -0.5609$ Å, $\theta = 177.3^\circ$, $\varphi = 18.6^\circ$; ring (C10–C15): $Q = 0.5655$ Å, $q_2 = 0.0111$ Å, $q_3 = -0.5654$ Å, $\theta = 178.8^\circ$, $\varphi = 198.1^\circ$. However, the 1,3-dioxane ring exhibits a boat configuration, with puckering parameters for (ring O1, C1, O2, C7–C9): $Q = 0.4927$ Å, $q_2 = 0.4914$ Å, $q_3 = -0.0363$ Å,

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.55198(6)	0.36931(8)	0.16144(8)	0.0466(2)
C1	0.50204(9)	0.42783(10)	0.25839(12)	0.0432(3)
O2	0.58017(7)	0.49863(8)	0.34824(10)	0.0580(3)
C2	0.45102(10)	0.34088(12)	0.33793(14)	0.0505(3)
H2A	0.424249	0.381584	0.408078	0.061*
H2B	0.505088	0.285214	0.382806	0.061*
C3	0.35853(11)	0.27613(14)	0.24412(18)	0.0676(4)
H3A	0.386945	0.227391	0.181669	0.081*
H3B	0.323821	0.225650	0.298594	0.081*
O3	0.68541(8)	0.26985(10)	0.11675(9)	0.0674(3)
O4	0.73949(9)	0.51692(11)	0.48470(13)	0.0896(4)
C4	0.27570(11)	0.35959(15)	0.16247(19)	0.0735(5)
H4A	0.242113	0.403447	0.224110	0.088*
H4B	0.219865	0.315734	0.100550	0.088*
C5	0.32927(12)	0.44313(14)	0.08118(16)	0.0660(4)
H5A	0.357643	0.399594	0.014404	0.079*
H5B	0.276102	0.497907	0.032166	0.079*
C6	0.41981(11)	0.50936(12)	0.17421(16)	0.0597(4)
H6A	0.454899	0.559145	0.119462	0.072*
H6B	0.390225	0.558902	0.234850	0.072*
C7	0.65165(9)	0.32385(10)	0.20039(12)	0.0426(3)
C8	0.71189(9)	0.34388(10)	0.34558(12)	0.0412(3)
H8	0.685559	0.283732	0.399187	0.049*
C9	0.68097(11)	0.45877(12)	0.39992(14)	0.0535(3)
C10	0.83495(9)	0.32619(11)	0.37526(13)	0.0472(3)
H10	0.863092	0.350099	0.470511	0.057*
C11	0.86677(11)	0.19880(12)	0.36705(15)	0.0590(4)
H11A	0.840900	0.170968	0.274029	0.071*
H11B	0.832908	0.152303	0.426199	0.071*
C12	0.98906(12)	0.18339(16)	0.41004(17)	0.0709(5)
H12A	1.013963	0.203399	0.506006	0.085*
H12B	1.007148	0.102289	0.398894	0.085*
C13	1.04564(13)	0.25955(18)	0.32569(19)	0.0820(5)
H13A	1.123089	0.252051	0.359378	0.098*
H13B	1.027364	0.233382	0.231296	0.098*
C14	1.01393(12)	0.38554(18)	0.3320(2)	0.0867(6)
H14A	1.048035	0.431160	0.272361	0.104*
H14B	1.040109	0.414104	0.424666	0.104*
C15	0.89133(11)	0.40296(13)	0.28907(17)	0.0638(4)
H15A	0.874069	0.484227	0.300773	0.077*
H15B	0.865785	0.383188	0.193129	0.077*

$\theta = 94.2^\circ$, $\varphi = 241.7^\circ$. There are weak intra- and intermolecular C—H...O interactions in the molecule.

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