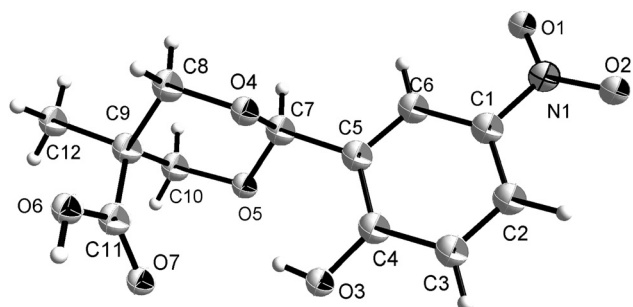


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Crystal structure of 2-(2-hydroxy-5-nitrophenyl)-5-methyl-1,3-dioxane-5-carboxylic acid, $C_{12}H_{13}N_1O_7$

**Table 1:** Data collection and handling.

Crystal:	Colourless block
Size:	$0.34 \times 0.28 \times 0.22$ mm
Wavelength:	MoK α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14162, 2914, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2398
$N(\text{param})_{\text{refined}}$:	182
Programs:	Bruker [1], Olex2 [2], SHELX [3]

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Abstract

$C_{12}H_{13}N_1O_7$, monoclinic, $P2_1/c$ (no. 14), $a = 11.416(7)$ Å, $b = 13.860(8)$ Å, $c = 8.215(5)$ Å, $\beta = 99.607(6)^\circ$, $V = 1281.6(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0433$, $wR_{\text{ref}}(F^2) = 0.1274$, $T = 293(2)$ 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

The mixture of 2,2-bis(hydroxymethyl)propionic acid (1.00 g, 7.455 mmol), 5-nitrosalicylaldehyde (1.49 g, 8.95 mmol), cyclohexane (15 mL), *N,N*-dimethylformamide (5 mL) and *p*-toluene sulfonic acid (0.01 g, 0.0058 mmol) were refluxed for 4 h. After the mixture was cooled, sodium bicarbonate (0.005 g, 0.0583 mmol) was added and stirred at room temperature for half an hour. The solvent was evaporated under reduced pressure and

then ethyl acetate was added to dissolve the residue. Subsequently, the solution was washed with brine (15 mL $\times$ 2) and water (15 mL $\times$ 2), respectively. Then the organic layer was dried with anhydrous sodium sulfate, filtered. The product was recrystallized from ethyl acetate to afford colorless crystals (1.2342 g, 4.3576 mmol; yield 58.45%).

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Acetal compounds are widely used in organic syntheses as a protection of carbonyl groups or intermediates [4, 5]. In addition, this type of compounds may have insecticidal as well as anti-foaming properties [6]. The crystal structures of some similar 1,3-dioxanes have been reported [7–10]. The new title compound was synthesized by 2,2-bis(hydroxymethyl)propionic acid, 5-nitrosalicylaldehyde and DMF, cyclohexane as solvent. *p*-toluenesulfonic acid as catalyst. The clear colourless crystal of the compound was obtained from ethyl acetate.

There is one molecule in the asymmetric unit of the title crystal structure (see the figure). All bond lengths and

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.58191 (13)	0.30364 (13)	0.7947 (2)	0.0993 (5)
O2	0.54109 (17)	0.44214 (14)	0.6758 (2)	0.1105 (6)
O3	0.11564 (10)	0.20329 (8)	0.31684 (14)	0.0647 (3)
H3	0.108172	0.145684	0.335370	0.097*
O4	0.31722 (8)	0.03745 (7)	0.35140 (11)	0.0458 (2)
O5	0.16717 (8)	0.04511 (6)	0.51097 (11)	0.0434 (2)
O6	0.12798 (11)	−0.08050 (9)	0.04965 (13)	0.0709 (4)
H6	0.081009	−0.056507	−0.025933	0.106*
O7	0.01778 (9)	−0.00054 (8)	0.20123 (12)	0.0571 (3)
N1	0.51989 (14)	0.35610 (14)	0.6939 (2)	0.0754 (5)
C1	0.41435 (13)	0.31572 (12)	0.59467 (18)	0.0558 (4)
C2	0.33457 (16)	0.37544 (11)	0.49927 (19)	0.0598 (4)
H2	0.348221	0.441528	0.496564	0.072*
C3	0.23443 (15)	0.33581 (11)	0.40811 (19)	0.0566 (4)
H3A	0.179024	0.375372	0.344279	0.068*
C4	0.21547 (12)	0.23684 (10)	0.41077 (16)	0.0476 (3)
C5	0.29839 (11)	0.17674 (10)	0.50589 (15)	0.0430 (3)
C6	0.39753 (12)	0.21735 (11)	0.59948 (17)	0.0496 (3)
H6A	0.452740	0.178638	0.665472	0.060*
C7	0.28636 (11)	0.06913 (10)	0.50181 (15)	0.0424 (3)
H7	0.339899	0.040476	0.594925	0.051*
C8	0.30764 (12)	−0.06515 (10)	0.33490 (17)	0.0472 (3)
H8A	0.361023	−0.095886	0.424110	0.057*
H8B	0.330449	−0.084929	0.231327	0.057*
C9	0.18072 (12)	−0.09671 (9)	0.33940 (16)	0.0452 (3)
C10	0.14764 (12)	−0.05691 (9)	0.49816 (16)	0.0461 (3)
H10A	0.064641	−0.070576	0.500797	0.055*
H10B	0.194873	−0.088757	0.591978	0.055*
C11	0.10011 (12)	−0.05399 (9)	0.19081 (16)	0.0451 (3)
C12	0.17036 (16)	−0.20683 (10)	0.3366 (2)	0.0616 (4)
H12A	0.089801	−0.225016	0.340967	0.092*
H12B	0.221967	−0.233300	0.430164	0.092*
H12C	0.192799	−0.231242	0.236832	0.092*

angles are in the expected ranges [8–11]. Two intramolecular hydrogen bonds of different strengths connect the hydroxy group (O3) with the acceptor atoms O5 and O7.

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