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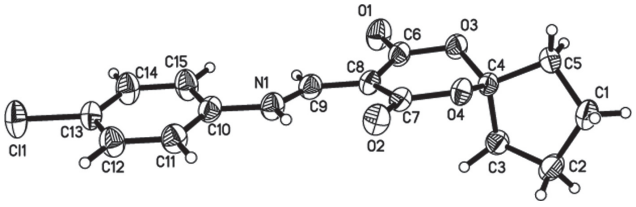
The crystal structure of 8-((4-chlorophenylamino) methylene)-6,10-dioxaspiro[4.5]decane-7,9-dione, $C_{15}H_{14}ClNO_4$



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Abstract

$C_{15}H_{14}ClNO_4$, monoclinic, $P2_1/n$ (no. 14), $a = 14.188(3)$ Å, $b = 5.6930(11)$ Å, $c = 17.444(4)$ Å, $\beta = 95.60(3)°$, $V = 1402.3(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0719$, $wR_{ref}(F^2) = 0.2214$, $T = 293(2)$ 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:	Colorless block
Size:	0.24 × 0.16 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.29 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, ω
θ_{max}, completeness:	27.5°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12280, 3178, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2026
$N(param)_{refined}$:	190
Programs:	Bruker [1], SHELX [2]

Source of material

The title compound was obtained following our reported method [3]. 6,10-dioxaspiro[4.5]decane-7,9-dione (1.70 g, 10 mmol) was dissoved in a solution of 95% ethanol (25 mL) and orthoformic acid methylester (1.27 g, 12 mmol) and stirred for 2 h at reflux. Then, 4-chlorobenzenamine (1.27 g, 10 mmol) was added. The solution was refluxed for another 5 h. The white precipitates were collected by filtration, washed three times and dried. Colorless block crystals of the title compound were obtained by evaporation from ethanol after a few days. The peak at 3213 cm^{-1} in the IR spectra (FT IR-650) is assigned to the N-CH stretching vibration. The other four peaks (1736, 1691, 1250, 1173 cm^{-1}) are due to the stretching vibrations of C=O and C—O groups.

Experimental details

All H atoms of $C_{15}H_{14}ClNO_4$ were fined with riding coordinates, and U_{iso}(H) is 1.2 times U_{eq}(C) atoms [2].

Comment

Recently, the synthesis of spiro compounds has drawn increased attention owing to their pharmacological and biological activities, e.g. spiro-pyranindol-2-one derivatives as immunomodulatory agents [4]. *N*,*N*-spiro bridged compounds having inhibitory effects on E. coli and B. cereus bacteria [5], and xanthene derivatives containing a spiro structure as potential antibacterial and anticholinesterases agents [6]. In addition spiro compounds are used as hole-transporting materials [7], in perovskite solar cells [8, 9], and in fluorescence devices [10, 11]. In our previous study, we had reported spiro compounds containing different groups, such as Meldrum's acid [12, 13], 1,5-dioxaspiro[5.5] undecane-2,4-dione [14, 15] and benzimidazole group [16].

As shown in the figure, there is one molecule in the asymmetric unit. The 6,10-dioxaspiro[4.5]decane-7,9-dione group connects the 4-chlorophenylamino group by atom C9, forming the N1—C9—C8 bond angle of 125.2(3)°. The bond length of N1—C9 (1.297(4) Å) and the bond length of C(9)—C(8) (1.388(4) Å) are similar to those of reported ones (N1—C10 (1.308(7) Å), C8—C10 (1.371(7) Å)) [17, 18]. The eight atoms

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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}
C1	0.0176(3)	−1.1856(6)	1.41346(17)	0.0775(10)
H1B	0.0741	−1.1412	1.4460	0.093*
H1C	−0.0199	−1.2895	1.4424	0.093*
C2	−0.0394(3)	−0.9685(6)	1.38767(17)	0.0700(9)
H2A	−0.1015	−0.9736	1.4065	0.084*
H2B	−0.0071	−0.8274	1.4072	0.084*
C3	−0.0486(2)	−0.9703(5)	1.30006(16)	0.0552(7)
H3A	−0.1077	−1.0423	1.2795	0.066*
H3B	−0.0453	−0.8124	1.2796	0.066*
C4	0.03569(18)	−1.1160(4)	1.28126(13)	0.0459(6)
C5	0.0443(2)	−1.3078(5)	1.34145(16)	0.0632(8)
H5A	0.1084	−1.3682	1.3489	0.076*
H5B	0.0012	−1.4364	1.3271	0.076*
C6	0.0364(2)	−1.0722(5)	1.14510(14)	0.0536(7)
C7	0.14182(18)	−0.8287(4)	1.23704(14)	0.0478(6)
C8	0.09430(19)	−0.8672(5)	1.16107(14)	0.0489(6)
C9	0.1117(2)	−0.7307(5)	1.09801(16)	0.0555(7)
H9A	0.0788	−0.7682	1.0508	0.067*
C10	0.1922(2)	−0.4157(5)	1.03517(15)	0.0555(7)
C11	0.2475(2)	−0.2228(5)	1.04970(16)	0.0619(7)
H11A	0.2692	−0.1835	1.1001	0.074*
C12	0.2713(2)	−0.0848(5)	0.98894(17)	0.0644(8)
H12A	0.3095	0.0468	0.9986	0.077*
C13	0.2386(2)	−0.1425(5)	0.91441(16)	0.0570(7)
C14	0.1824(3)	−0.3337(6)	0.89930(17)	0.0698(9)
H14A	0.1603	−0.3708	0.8488	0.084*
C15	0.1581(3)	−0.4745(5)	0.96027(18)	0.0687(9)
H15A	0.1196	−0.6056	0.9507	0.082*
Cl1	0.27056(7)	0.03026(17)	0.83858(5)	0.0821(4)
N1	0.17008(17)	−0.5551(4)	1.09963(13)	0.0561(6)
H1A	0.1984	−0.5183	1.1438	0.067*
O1	0.00191(18)	−1.1385(4)	1.08227(11)	0.0766(7)
O2	0.20019(15)	−0.6786(4)	1.25352(11)	0.0626(6)
O3	0.02464(14)	−1.2147(3)	1.20621(10)	0.0573(5)
O4	0.12003(12)	−0.9780(3)	1.29342(10)	0.0507(5)

(Cl1, N1, C10—C15) of 4-chlorophenylamino ring and the four atoms O1, C6, C8, C9 are co-planar, where the maximum deviation is 0.037(2) Å. N(1)—H(1A)···O(2) interactions appeared in the molecule which can stabilize the structure.

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