Crystal structure of cesium 4,6-dinitroresorcinolate, $CsC_6H_3O_2(NO_3)_2$

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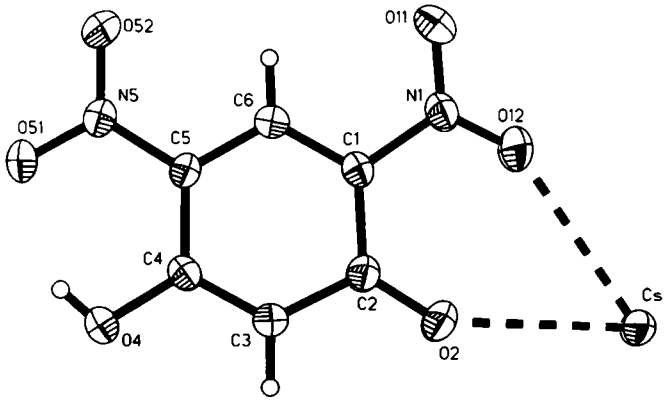

Abstract

$C_6H_3CsN_2O_6$, triclinic, $P\bar{1}$ (No. 2), $a = 7.114(2)$ Å, $b = 7.572(1)$ Å, $c = 9.314(2)$ Å, $\alpha = 92.18(1)°$, $\beta = 111.82(2)°$, $\gamma = 107.64(2)°$, $V = 437.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.019$, $wR_{ref}(F^2) = 0.052$, $T = 291$ K.

Source of material

The starting compound 4,6-dinitroresorcinol was synthesized according the procedure described by Beyerlein [1]. The title compound was obtained by neutralizing a water-ethanolic 1:1 solution of 4,6-dinitroresorcinol with cesium carbonate (Cs_2CO_3 99.8% Aldrich) in a 2:1 molar ratio at room temperature by continuous stirring for 40 minutes. The resulting cesium 4,6-dinitroresorcinolate was isolated and recrystallized twice from ethanol-water and after that from doubly distilled water. Over 530 K the compound decomposes without melting. The purity of the compound was confirmed by elemental analysis, IR, UV-vis and mass spectrometry. Red transparent crystals, suitable for X-ray diffraction were grown from solution of doubly distilled water by slow evaporation over period of two weeks.

Discussion

Organic nonlinear optical (NLO) materials have attracted much attention in the past years due to potential applications in various fields such as telecommunication, optical data storage and optical information processing. Because of their notable chemical flexibility that allows for molecular engineering of the nonlinear optical responses and their fast electronic responses, organic materials are particularly interesting candidates for the elaboration of optimized NLO materials [2, 3, 4].

The conversion of hydroxy group of 4,6-dinitroresorcinol into the corresponding oxo-anion is a way to enhance the charge transfer transition on the molecular level - a requisite for a design of efficient second- and third-order nonlinear optical materials.

Table 1. Data collection and handling.

Crystal:	orange parallelepiped, size 0.2 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	42.46 cm^{-1}
Diffractometer, scan mode:	Nonius MACH3, 2θ/ω
$2\theta_{max}$:	54.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2161, 1999
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1933
$N(param)_{refined}$:	149
Programs:	PARST95 [5], SHELXS-97 [6], SHELXTL [7], SHELXL-97 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.293(5)	0.786(5)	0.033(4)	0.036(8)
H(6)	2*i*	0.216(5)	0.184(5)	0.135(4)	0.035(8)
H(4)	2*i*	0.233(8)	0.486(7)	–0.248(6)	0.07(2)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cs	2*i*	0.27477(3)	0.88625(2)	0.59453(2)	0.0579(1)	0.0266(1)	0.0330(1)	0.01348(8)	0.02787(9)	0.00514(7)
C(1)	2*i*	0.2554(4)	0.4389(3)	0.2312(3)	0.030(1)	0.027(1)	0.022(1)	0.0093(9)	0.0128(9)	0.0057(9)
C(2)	2*i*	0.2863(4)	0.6333(3)	0.2077(3)	0.032(1)	0.026(1)	0.025(1)	0.0082(9)	0.0141(9)	0.0023(9)
C(3)	2*i*	0.2836(4)	0.6650(4)	0.0560(3)	0.037(1)	0.023(1)	0.028(1)	0.011(1)	0.016(1)	0.0059(9)
C(4)	2*i*	0.2509(4)	0.5283(3)	–0.0586(3)	0.029(1)	0.026(1)	0.023(1)	0.0087(9)	0.0122(9)	0.0066(9)
C(5)	2*i*	0.2241(4)	0.3410(3)	–0.0268(3)	0.031(1)	0.023(1)	0.024(1)	0.0103(9)	0.0144(9)	0.0024(8)
C(6)	2*i*	0.2286(4)	0.3017(3)	0.1181(3)	0.032(1)	0.023(1)	0.025(1)	0.0105(9)	0.0123(9)	0.0060(9)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.2432(4)	0.3780(3)	0.3736(2)	0.038(1)	0.032(1)	0.023(1)	0.0094(9)	0.0142(8)	0.0063(8)
O(11)	2*i*	0.2464(6)	0.2200(4)	0.3958(3)	0.119(2)	0.043(1)	0.040(1)	0.037(1)	0.042(1)	0.023(1)
O(12)	2*i*	0.2248(5)	0.4804(3)	0.4674(3)	0.093(2)	0.045(1)	0.036(1)	0.025(1)	0.043(1)	0.0088(9)
O(2)	2*i*	0.3133(4)	0.7644(3)	0.3061(2)	0.065(1)	0.028(1)	0.036(1)	0.0117(9)	0.029(1)	–0.0007(8)
O(4)	2*i*	0.2468(4)	0.5741(3)	–0.1978(2)	0.058(1)	0.031(1)	0.0261(9)	0.0161(9)	0.0223(9)	0.0095(8)
N(5)	2*i*	0.1929(4)	0.1937(3)	–0.1387(3)	0.042(1)	0.028(1)	0.028(1)	0.0118(9)	0.0179(9)	0.0027(8)
O(51)	2*i*	0.1730(4)	0.2236(3)	–0.2747(2)	0.063(1)	0.038(1)	0.0257(9)	0.0195(9)	0.0243(9)	0.0042(8)
O(52)	2*i*	0.1885(5)	0.0378(3)	–0.1041(3)	0.091(2)	0.028(1)	0.046(1)	0.026(1)	0.040(1)	0.0094(9)

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