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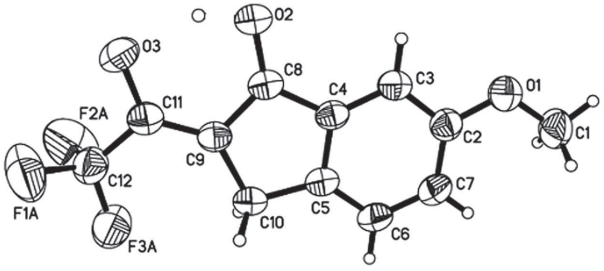
Crystal structure of (*Z*)-6-methoxy-2-(2,2,2-trifluoro-1-hydroxyethylidene)-2,3-dihydro-1*H*-inden-1-one, $C_{12}H_6F_6O_3$



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Abstract

$C_{12}H_6F_6O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 9.433(1)$ Å, $b = 15.567(2)$ Å, $c = 7.682(1)$ Å, $\beta = 102.83(1)°$, $V = 1099.95(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0433$, $wR_{ref}(F^2) = 0.1264$, $T = 293$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless blocks
Size:	0.36 × 0.36 × 0.32 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.4 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	56.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9317, 2746, 0.020
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2218
$N(param)_{refined}$:	194
Programs:	SHELX [3], Bruker programs [7, 8]

Source of material

The title compound was synthesised following a literature method [1, 2]. In a typical reaction, to a solution of 6-methoxy-2,3-dihydro-1*H*-inden-1-one (2.5 g, 11.57 mmol) in 60 mL dry tetrahydrofuran, ethyl 2,2,2-trifluoroacetate (2.46 g, 17.31 mmol) was added, and the mixture was allowed to stir for 45 min. Then NaH (0.555 g, 13.88 mmol) was added, and the mixture was allowed to stir for 48 h at room temperature under a N_2 atmosphere. The resulting mixture was poured into 100 mL water and acidified to pH 2–3 using 2 N hydrochloric acid. Then the mixture was extracted with dichloromethane (3 × 10 mL) and the combined organic extracts were dried with anhydrous Na_2SO_4. The solvent was removed by rotary evaporation and the yellow residue was dried under vacuum. The blocked-shaped crystals of the title compound were obtained in about 5 days by recrystallization from hexane.

Experimental details

All H atoms on C atoms were placed in idealized positions [C—H = 0.97 (methylene) and 0.93 Å(aromatic)] and included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$(methylene and aromatic C). The title structure was solved by direct methods and refined by full-matrix least-squares on F^2 using the SHELXTL program package [3]. The trifluoromethyl group shows disorder which is for clarity not shown in the figure.

Discussion

β-Diketonate ligands have been widely used to construct different lanthanide complexes for luminescence properties or precursors of metalorganic chemical vapor deposition for single and multi-component oxide thin films [4–6]. Herein,

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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	0.1693(2)	0.41034(13)	0.9411(3)	0.0669(5)
H1A	0.0965	0.4322	0.8435	0.100*
H1B	0.1550	0.4344	1.0510	0.100*
H1C	0.1618	0.3489	0.9453	0.100*
C2	0.35259(16)	0.40597(10)	0.76770(19)	0.0461(3)
C3	0.49268(15)	0.42892(9)	0.75802(19)	0.0441(3)
H3	0.5518	0.4604	0.8489	0.053*
C4	0.54196(14)	0.40363(8)	0.60921(17)	0.0389(3)
C5	0.45637(15)	0.35642(8)	0.46937(18)	0.0411(3)
C6	0.31607(16)	0.33473(10)	0.4811(2)	0.0496(4)
H6	0.2566	0.3038	0.3897	0.060*
C7	0.26481(16)	0.35920(10)	0.6292(2)	0.0507(4)
H7	0.1708	0.3444	0.6367	0.061*
C8	0.68181(14)	0.42003(8)	0.56435(18)	0.0407(3)
C9	0.68290(14)	0.37981(8)	0.39622(17)	0.0397(3)
C10	0.53657(15)	0.33791(9)	0.32407(18)	0.0439(3)
H10A	0.4877	0.3635	0.2116	0.053*
H10B	0.5466	0.2766	0.3077	0.053*
C11	0.80797(16)	0.38173(9)	0.3348(2)	0.0463(3)
C12	0.82823(18)	0.33243(12)	0.1727(2)	0.0583(4)
F1A[a]	0.8735(4)	0.3860(2)	0.0571(5)	0.0928(10)
F2A[a]	0.9230(5)	0.2734(3)	0.2084(7)	0.1201(19)
F3A[a]	0.7086(4)	0.3005(3)	0.0776(6)	0.0933(14)
F1B[b]	0.9352(7)	0.3528(6)	0.1105(10)	0.122(3)
F2B[b]	0.8580(10)	0.2493(5)	0.2308(12)	0.123(3)
F3B[b]	0.7113(8)	0.3232(6)	0.0532(10)	0.103(3)
O1	0.30946(13)	0.43341(9)	0.91686(16)	0.0628(3)
O2	0.78655(11)	0.46182(8)	0.65983(14)	0.0549(3)
O3	0.92443(12)	0.42328(8)	0.41377(17)	0.0634(3)
H2-3	0.865(2)	0.4601(16)	0.588(3)	0.095*

[a]Occupancy: 0.64; [b]Occupancy: 0.36.

we report the crystal structure of the *β*-diketone, (*Z*)-6-methoxy-2-(2,2,2-trifluoro-1-hydroxyethylidene)-2,3-dihydro-1*H*-inden-1-one. The structural analysis reveals that almost all carbon atoms in the molecule are coplanar. The bond lengths and bond angles are all in normal ranges. The crystal structure features an intramolecular O—H···O hydrogen bond (*cf*. the figure; O2···O3) The hydrogen atom of the formal hydroxy group is more or less centered between the oxygen atoms. The resolution of the routine diffraction data does not allow the refinement of a probable double minimum potential. According to the O···O distances of 2.595(2) Å this hydrogen bond is strong.

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