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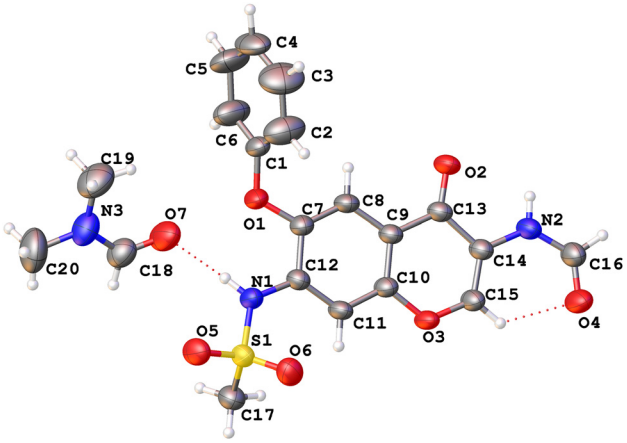
The crystal structure of iguratimod-dimethylformamide (1/1), $C_{17}H_{14}N_2O_6S{\cdot}C_3H_7NO$



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Abstract

$C_{17}H_{14}N_2O_6S{\cdot}C_3H_7NO$, triclinic, $P\bar{1}$ (no. 2), a = 5.4146(4) Å, b = 11.7925(8) Å, c = 16.9527(12) Å, α = 85.238(2)°, β = 86.674(4)°, γ = 78.959(2)°, V = 1057.77(13) Å^3, Z = 2, $R_{gt}(F)$ = 0.0418, $wR_{ref}(F^2)$ = 0.1175, T = 296(2) K.

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The asymmetric unit of the title crystal 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.

Table 1: Data collection and handling.

Crystal:	Needle
Size:	0.24 × 0.22 × 0.20 mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ:	1.78 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-AXS, φ and ω
θ_{max}, completeness:	68.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	24,753, 3854, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3546
$N(param)_{refined}$:	288
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

1 Source of material

In representative experiments, iguratimod was presented by Jiangsu Zhengda Qingjiang Pharmaceutical Co. with no further purification. The iguratimod was dissolved in *N*,*N*-dimethylformamide (DMF), and the solution was filtered and placed in a vial, covered with membrane and punctured. The filtrate was slowly evaporated at room temperature to obtain crystals of the title compound.

2 Experimental details

Using Olex2 [2], the structure was solved with the XT [3] structure solution program and refined with the ShelXL [4] refinement package. All H atoms were positioned geometrically and treated as riding on their parent atoms, with the d(C–H) = 0.96 Å (methyl) and d(C–H) = 0.93 Å (aromatic), and U_{iso}(H) = 1.2 times U_{iso}(C) and U_{iso}(H) = 1.5 times U_{iso}(O).

3 Comment

Iguratimod is known for its anti-inflammatory activities and therapeutic effects in patients with rheumatoid arthritis, which can significantly inhibit the initiation and progression of rheumatoid arthritis by regulating T cell differentiation, reducing the production of pro-inflammatory cytokines and immunoglobulins, promoting bone formation, and inhibiting bone resorption [5, 6]. The α and β crystal forms of iguratimod were prepared and five crystal forms of iguratimod were obtained with ethanol, acetonitrile, dimethylformamide, acetone, and dichloromethane [7, 8]. However, the single crystal structure of iguratimod-DMF has not been reported.

The asymmetric unit of the title structure contains one iguratimod molecule and one dimethylformamide molecule (see figure). It indicates that the hydrogen bond

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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	0.0426 (4)	0.42521 (15)	0.77370 (12)	0.0510 (5)
C2	0.2254 (6)	0.4435 (2)	0.81997 (19)	0.0925 (9)
H2A	0.366857	0.386247	0.829180	0.111*
C3	0.1980 (8)	0.5487 (3)	0.8533 (2)	0.1105 (11)
H3	0.319780	0.561948	0.886072	0.133*
C4	−0.0099 (7)	0.6341 (2)	0.83781 (18)	0.0904 (9)
H4	−0.031668	0.703940	0.861402	0.108*
C5	−0.1779 (6)	0.6155 (2)	0.7891 (2)	0.1015 (10)
H5	−0.314086	0.674260	0.776889	0.122*
C6	−0.1550 (5)	0.5100 (2)	0.75590 (19)	0.0852 (8)
H6	−0.274621	0.498092	0.721811	0.102*
C7	0.2599 (3)	0.27032 (15)	0.69920 (11)	0.0454 (4)
C8	0.4074 (3)	0.33257 (14)	0.65258 (11)	0.0474 (4)
H8	0.373950	0.413004	0.651693	0.057*
C9	0.6091 (3)	0.27654 (14)	0.60597 (10)	0.0421 (4)
C10	0.6438 (3)	0.15677 (14)	0.60668 (10)	0.0439 (4)
C11	0.4935 (3)	0.09182 (14)	0.65300 (11)	0.0455 (4)
H11	0.520180	0.011807	0.650997	0.055*
C12	0.3047 (3)	0.14721 (14)	0.70189 (10)	0.0423 (4)
C13	0.7776 (3)	0.33921 (14)	0.55821 (11)	0.0444 (4)
C14	0.9729 (3)	0.26731 (14)	0.51228 (10)	0.0439 (4)
C15	0.9908 (4)	0.15174 (15)	0.51637 (11)	0.0507 (5)
H15	1.118352	0.108539	0.486260	0.061*
C16	1.3399 (4)	0.27983 (17)	0.42287 (12)	0.0575 (5)
H16	1.436555	0.330208	0.397269	0.069*
C17	0.0927 (4)	−0.11520 (18)	0.70668 (13)	0.0596 (5)
H17A	0.172147	−0.094876	0.656775	0.089*
H17B	−0.086206	−0.089607	0.704442	0.089*
H17C	0.130014	−0.197737	0.717597	0.089*
C18	−0.3941 (4)	0.1554 (2)	0.89121 (13)	0.0665 (6)
H18	−0.433763	0.092469	0.868432	0.080*
C19	−0.4664 (6)	0.2825 (3)	0.99769 (18)	0.0997 (10)
H19A	−0.410290	0.253499	1.049486	0.150*
H19B	−0.615328	0.340977	1.002542	0.150*
H19C	−0.336438	0.315320	0.968512	0.150*
C20	−0.7255 (6)	0.1334 (4)	0.9900 (2)	0.1188 (12)
H20A	−0.882776	0.186642	0.985071	0.178*
H20B	−0.698289	0.110485	1.044952	0.178*
H20C	−0.729885	0.066215	0.962117	0.178*
N1	0.1510 (3)	0.09013 (12)	0.75419 (9)	0.0497 (4)
H1	0.016179	0.131409	0.773472	0.060*
N2	1.1380 (3)	0.32644 (13)	0.46624 (10)	0.0510 (4)
H2	1.110 (4)	0.398 (2)	0.4685 (14)	0.071 (7)*
N3	−0.5220 (4)	0.18898 (18)	0.95645 (11)	0.0696 (5)
O1	0.0523 (3)	0.31806 (11)	0.74362 (10)	0.0650 (4)
O2	0.7611 (3)	0.44536 (10)	0.55664 (9)	0.0613 (4)
O3	0.8333 (3)	0.09479 (10)	0.56188 (8)	0.0537 (4)
O4	1.4084 (3)	0.17708 (13)	0.41397 (10)	0.0767 (5)
O5	0.0539 (3)	−0.06048 (12)	0.85205 (9)	0.0659 (4)
O6	0.4714 (3)	−0.08905 (12)	0.78444 (9)	0.0602 (4)
O7	−0.2292 (4)	0.20068 (15)	0.85890 (12)	0.0926 (6)
S1	0.20612 (8)	−0.04847 (3)	0.78176 (3)	0.04649 (18)

plays an important role in maintaining the crystal structure. The O7 atom on the amide group of dimethylformamide is a hydrogen bond acceptor, and the H1 atom on the sulfonamide (N1–H1) of iguratimod is a hydrogen bond donor, forming the intermolecular hydrogen bond N1–H1···O7 [length 2.832(2) Å, angle 156.75°]. The O atom on the formamide groups of iguratimod is a hydrogen bond acceptor. The H15 atom on the benzopyran rings (C15–H15) of iguratimod is hydrogen bond donor, forming the intramolecular hydrogen bond C15–H15···O4 [length 2.817 Å, angle 125.54°]. The O atom on the formamide groups of iguratimod is a hydrogen bond acceptor. The H15 atom on the benzopyran rings (N2–H2) of iguratimod is hydrogen bond donor, forming the intramolecular hydrogen bond N2–H2···O2 [length 2.838(2) Å, angle 147.66°]. All bond lengths and angles are within a reasonable range [9].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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