

Methyl (*Z,Z*)- α -(methoxyimino)-2-[(1-[3-(trifluoromethyl)phenyl]ethylidene)amino]oxy-methyl]benzeneacetate

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Key indicators

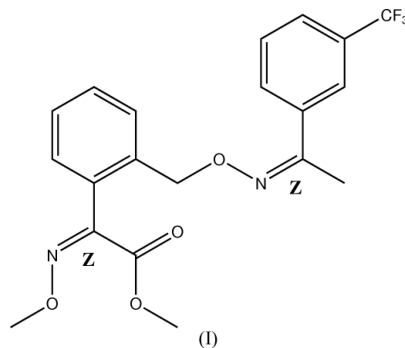
Single-crystal X-ray study
 $T = 291$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 Disorder in main residue
 R factor = 0.034
 wR factor = 0.069
 Data-to-parameter ratio = 12.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $\text{C}_{20}\text{H}_{19}\text{F}_3\text{N}_2\text{O}_4$, contains one molecule in the asymmetric unit. The CF_3 group is disordered. Important features of the molecule are the two $\text{C}=\text{N}$ bonds in a *Z,Z* configuration, one in the β -methoxy-system and the other in the oxymethyl side-chain between the two aromatic rings.

Comment

The title compound, (I), is an isomer of the fungicide trifloxystrobin (TFS). TFS appears in an *E,E* configuration (Ebeling *et al.*, 2003) but, on exposure to the environment, it is susceptible to conversion to a mixture of four isomers. As part of our investigations of the isomerization of TFS in the environment, we have determined the crystal structure of (I). The general synthesis route of TFS was described by Ziegler *et al.* (2003).



Experimental

Compound (I) was produced by illuminating a solution of TFS (200 mg) in acetone (50 ml) in a Suntest apparatus at 500 W m^{-2} in a quartz vessel. Compound (I) appeared as a component of a mixture of isomers of TFS and was identified by comparison with the chromatogram of the reference standard obtained from Bayer Crop Science, Germany. Compound (I) was isolated from the mixture of isomers by preparative high-pressure liquid chromatography (HPLC), employing a Gynkotek HPLC equipped with a preparative Kromasil 100 C18 column ($7 \mu\text{m}$, $250 \times 20 \text{ mm}$) and a UV detector set at 250 nm. The mobile phase was acetonitrile–water (90:10 *v/v*) at a rate 4 ml min^{-1} . Compound (I) eluted with a good baseline separation at a retention time of 21.81 min. The fraction corresponding to (I) was collected separately and evaporated to complete dryness at 303 K under vacuum. The purity of the white solid thus obtained was confirmed by IR, Raman, NMR and mass spectrometry. It was redissolved in a minimum quantity of methanol and kept inside a fume-hood at room temperature for slow evaporation. Compound (I) crystallized out over a period of a week.

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Crystal data

$C_{20}H_{19}F_3N_2O_4$
 $M_r = 408.37$
 Monoclinic, $P2_1/n$
 $a = 8.0122$ (11) Å
 $b = 6.9993$ (6) Å
 $c = 36.417$ (4) Å
 $\beta = 91.945$ (5)°
 $V = 2041.1$ (4) Å³
 $Z = 4$

$D_x = 1.329$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 16 494 reflections
 $\theta = 3.0$ – 25.1 °
 $\mu = 0.11$ mm⁻¹
 $T = 291$ (1) K
 Block, colourless
 $0.20 \times 0.15 \times 0.15$ mm

Data collection

Nonius KappaCCD diffractometer
 ω scans
 16 494 measured reflections
 3538 independent reflections
 1275 reflections with $I > 2\sigma(I)$

$R_{int} = 0.038$
 $\theta_{max} = 25.1$ °
 $h = -9 \rightarrow 9$
 $k = -8 \rightarrow 8$
 $l = -43 \rightarrow 43$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.069$
 $S = 1.01$
 3538 reflections
 291 parameters

H-atom parameters constrained
 $w = \exp[4.20(\sin\theta/\lambda)^2]/[\sigma^2(F_o^2)]$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.09$ e Å⁻³
 $\Delta\rho_{min} = -0.13$ e Å⁻³

H atoms were placed in calculated positions, with C–H = 0.93–0.97 Å, and were treated as riding, with $U_{iso} = 1.5U_{eq}(C)$ for methyl groups and $1.2U_{eq}(C)$ for others; methyl groups C9 and C19 were allowed to rotate but not to tip, whereas methyl group C10 was positioned purely geometrically. Each F atom is disordered over two positions with an occupancy of 0.5.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2001).

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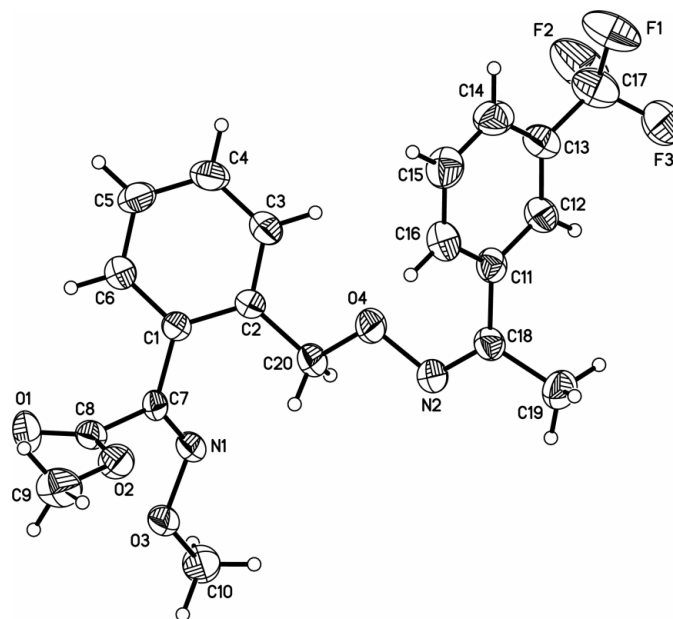


Figure 1

The molecular structure of (I), showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 30% probability level. The F atoms are disordered, and for each F atom, only one of the two split positions is shown.

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