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Ethyl (*E*)-3-anilino-2-cyano-3-mercapto-acrylate

Yong-Qi Qin, Fang-Fang Jian,* Ming-Na Jiang and Xiao-Yan Ren

New Materials and Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China
Correspondence e-mail: ffj2003@163169.net

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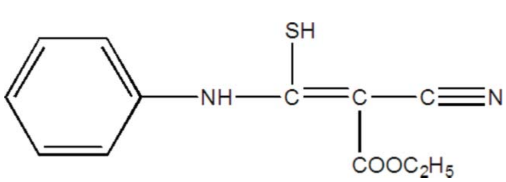
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.104; data-to-parameter ratio = 16.9.

In the title compound, $C_{12}H_{12}N_2O_2S$, there are S—H···N and N—H···O hydrogen-bond interactions. The N—H···O hydrogen bond is bifurcated, with the hydrogen being simultaneously donated to two equivalent O atoms, forming one intra- and one intermolecular N—H···O bond with an $R_1^2(4)$ motif. The motif of the S—H···N hydrogen bond is $R_2^2(12)$.

Related literature

For related literature, see: Allen (2002); Azim *et al.* (1997); Gao *et al.* (2006); Timofeeva *et al.* (2004); Xue *et al.* (2004); Etter *et al.* (1990).



Experimental

Crystal data

$C_{12}H_{12}N_2O_2S$
M_r = 248.30
Monoclinic, $C2/c$
a = 26.357 (5) Å
b = 7.0120 (14) Å
c = 16.234 (3) Å
β = 121.45 (3)°
V = 2559.6 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 295 (2) K
0.2 × 0.15 × 0.11 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
5482 measured reflections
2779 independent reflections
1979 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
3 standard reflections every 100 reflections intensity decay: 4.2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.104
S = 1.03
2779 reflections
164 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.83 (2)	2.05 (2)	2.7210 (19)	137.9 (19)
N2—H2A···O1^{i}	0.83 (2)	2.54 (2)	3.1513 (19)	131.3 (18)
S1—H1···N1^{ii}	1.20 (2)	2.45 (2)	3.4560 (17)	140.1 (15)

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x, -y+1, -z+1$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL/PC* (Sheldrick, 1997*b*); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2066).

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supplementary materials

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Ethyl (*E*)-3-anilino-2-cyano-3-mercaptoacrylate

Y.-Q. Qin, F.-F. Jian, M.-N. Jiang and X.-Y. Ren

Comment

Acrylics have been studied for many years because of their special chemical properties. They are widely used as elastics, adhesives, covering material and so on. Recent studies have also shown that the derivative of acrylics provide also herbicidal activity (Gao *et al.*, 2006).

It follows from our previous quantum-mechanical study of these compounds that they have several active centres and can easily form polyligand complexes with metals (Xue *et al.*, 2004).

In order to search for new compounds with higher bioactivity, the title compound was synthesized.

The C≡N bond length (1.145 (2) Å), C═C (1.405 (2) Å) and C═O (1.2202 (18) Å) are in agreement with those observed before (Timofeeva *et al.*, 2004; Azim *et al.*, 1997). The S—H hydrogen bond length corresponds well to the the value 1.197 (9) Å from 247 observations yielded by the Cambridge Crystallographic Database (Allen, 2002).

The H2A hydrogen is simultaneously donated to two equivalent O atoms, forming one intra- and one intermolecular N—H···O bond with a motif $R_1^2(4)$ (Etter *et al.*, 1990). A motif of the S—H···N hydrogen bond is $R_2^2(12)$.

Experimental

The title compound was prepared by the reaction of ethyl 2-cyanoacetate (0.02 mol), KOH (0.03 mol) and *N*-phenylmethanethioamide (0.02 mol) dissolved in 1,4-dioxane (30 ml) while refluxing about two hours. Yellow single crystals of suitable for X-ray measurements were prisms and they were obtained by recrystallization from ethanol/acetone (1:1 *v*/*v*) at room temperature that took about two days. The size of the crystals was about tenths of milimetres in each direction.

Refinement

All the H atoms were discernible in a difference Fourier map. The C—H distances were constrained to 0.93, 0.97 and 0.96 Å for the aryl, methylene and the methyl H atoms, respectively, while U_{iso}(H) = 1.2U_{eq}(C) for the aryls as well as for the methylenes and 1.5U_{eq}(C) for the methyls. The positional parameters as well as the U_{iso} of the H atoms involved in the S—H···N and N—H···O hydrogen bonds were refined freely.

Figures

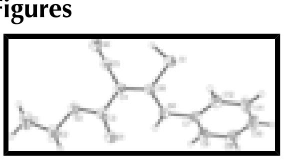
Fig. 1. The molecular structure and atom-labelling scheme of the title structure with the displacement ellipsoids drawn at the 30% probability level.

Ethyl *(E)*-3-anilino-2-cyano-3-mercaptoacrylate

Crystal data

$C_{12}H_{12}N_2O_2S$	F_{000} = 1040
M_r = 248.30	D_x = 1.289 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 221.3 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 26.357 (5) Å	Cell parameters from 25 reflections
b = 7.0120 (14) Å	θ = 1.8–27.0º
c = 16.234 (3) Å	μ = 0.24 mm^{-1}
β = 121.45 (3)º	*T* = 295 (2) K
V = 2559.6 (9) Å^3	Prism, yellow
Z = 8	0.2 × 0.15 × 0.11 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.019
Radiation source: fine-focus sealed tube	θ_{max} = 27.0º
Monochromator: graphite	θ_{min} = 1.8º
T = 295(2) K	*h* = −33→32
ω scan	*k* = −8→0
Absorption correction: none	*l* = −20→20
5482 measured reflections	3 standard reflections
2779 independent reflections	every 100 reflections
1979 reflections with *I* > 2σ(*I*)	intensity decay: 4.2%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.036	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.104	$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.5262P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ < 0.001
2779 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
164 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
37 constraints	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0029 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.036112 (18)	0.79399 (8)	0.39148 (4)	0.05947 (18)
O1	0.23012 (5)	0.56216 (18)	0.53519 (9)	0.0582 (3)
O2	0.20130 (5)	0.32520 (16)	0.59661 (8)	0.0494 (3)
N1	0.06376 (7)	0.3767 (2)	0.54908 (13)	0.0659 (4)
N2	0.14258 (6)	0.8090 (2)	0.41610 (10)	0.0462 (3)
H2A	0.1777 (10)	0.773 (3)	0.4423 (16)	0.069 (6)*
C1	0.25743 (12)	0.0566 (4)	0.6871 (2)	0.0964 (8)
H1A	0.2955	−0.0050	0.7170	0.145*
H1B	0.2274	−0.0285	0.6414	0.145*
H1C	0.2486	0.0903	0.7357	0.145*
C2	0.25859 (8)	0.2327 (3)	0.63637 (15)	0.0616 (5)
H2B	0.2901	0.3172	0.6813	0.074*
H2C	0.2657	0.1999	0.5851	0.074*
C3	0.19241 (7)	0.4885 (2)	0.54693 (11)	0.0421 (4)
C4	0.13246 (6)	0.5611 (2)	0.50890 (10)	0.0412 (3)
C5	0.09459 (7)	0.4589 (2)	0.53170 (12)	0.0461 (4)
C6	0.10985 (6)	0.7154 (2)	0.44426 (11)	0.0398 (3)
C7	0.11984 (6)	0.9531 (2)	0.34181 (11)	0.0420 (4)
C8	0.11445 (7)	0.9104 (3)	0.25486 (12)	0.0528 (4)
H8A	0.1259	0.7914	0.2449	0.063*
C9	0.09161 (9)	1.0473 (3)	0.18194 (14)	0.0660 (5)
H9A	0.0881	1.0201	0.1231	0.079*
C10	0.07420 (9)	1.2232 (3)	0.19681 (16)	0.0705 (6)
H10A	0.0584	1.3133	0.1475	0.085*
C11	0.08016 (9)	1.2657 (3)	0.28392 (17)	0.0679 (5)
H11A	0.0685	1.3846	0.2935	0.081*
C12	0.10358 (8)	1.1319 (3)	0.35789 (13)	0.0550 (4)
H12A	0.1083	1.1614	0.4174	0.066*
H1	0.0213 (9)	0.687 (3)	0.4325 (16)	0.085 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0395 (2)	0.0700 (3)	0.0704 (3)	0.0091 (2)	0.0297 (2)	0.0194 (2)
O1	0.0449 (6)	0.0577 (7)	0.0779 (8)	0.0069 (6)	0.0361 (6)	0.0216 (6)
O2	0.0457 (6)	0.0456 (6)	0.0564 (7)	0.0061 (5)	0.0262 (5)	0.0135 (5)
N1	0.0641 (9)	0.0628 (10)	0.0857 (11)	0.0043 (8)	0.0495 (9)	0.0187 (9)
N2	0.0372 (7)	0.0493 (8)	0.0532 (8)	0.0046 (6)	0.0245 (6)	0.0133 (6)
C1	0.0896 (16)	0.0770 (16)	0.118 (2)	0.0293 (14)	0.0511 (15)	0.0501 (15)
C2	0.0547 (10)	0.0575 (11)	0.0718 (12)	0.0167 (9)	0.0325 (9)	0.0176 (9)
C3	0.0435 (8)	0.0415 (8)	0.0420 (8)	0.0002 (7)	0.0227 (7)	0.0022 (7)
C4	0.0409 (8)	0.0415 (8)	0.0449 (8)	−0.0004 (7)	0.0249 (7)	0.0019 (7)
C5	0.0466 (8)	0.0453 (9)	0.0522 (9)	0.0046 (7)	0.0298 (7)	0.0065 (7)
C6	0.0366 (7)	0.0426 (8)	0.0413 (8)	−0.0005 (6)	0.0211 (6)	−0.0012 (7)
C7	0.0346 (7)	0.0426 (9)	0.0468 (8)	−0.0018 (6)	0.0198 (7)	0.0051 (7)
C8	0.0529 (9)	0.0502 (10)	0.0566 (10)	−0.0034 (8)	0.0294 (8)	−0.0005 (8)
C9	0.0662 (11)	0.0787 (14)	0.0475 (10)	−0.0107 (11)	0.0257 (9)	0.0072 (10)
C10	0.0604 (11)	0.0656 (13)	0.0694 (13)	0.0022 (10)	0.0227 (10)	0.0297 (11)
C11	0.0689 (12)	0.0444 (10)	0.0863 (15)	0.0096 (9)	0.0376 (11)	0.0140 (10)
C12	0.0603 (10)	0.0471 (10)	0.0601 (11)	0.0011 (8)	0.0332 (9)	0.0001 (8)

Geometric parameters (Å, °)

S1—C6	1.7544 (16)	C3—C4	1.456 (2)
S1—H1	1.20 (2)	C4—C6	1.405 (2)
O1—C3	1.2202 (18)	C4—C5	1.426 (2)
O2—C3	1.3485 (18)	C7—C8	1.376 (2)
O2—C2	1.449 (2)	C7—C12	1.393 (2)
N1—C5	1.145 (2)	C8—C9	1.394 (3)
N2—C6	1.3400 (19)	C8—H8A	0.9300
N2—C7	1.442 (2)	C9—C10	1.381 (3)
N2—H2A	0.83 (2)	C9—H9A	0.9300
C1—C2	1.493 (3)	C10—C11	1.372 (3)
C1—H1A	0.9600	C10—H10A	0.9300
C1—H1B	0.9600	C11—C12	1.389 (3)
C1—H1C	0.9600	C11—H11A	0.9300
C2—H2B	0.9700	C12—H12A	0.9300
C2—H2C	0.9700		
C6—S1—H1	97.5 (10)	N1—C5—C4	179.3 (2)
C3—O2—C2	117.53 (12)	N2—C6—C4	122.31 (13)
C6—N2—C7	124.61 (13)	N2—C6—S1	115.19 (12)
C6—N2—H2A	114.7 (14)	C4—C6—S1	122.47 (11)
C7—N2—H2A	120.6 (15)	C8—C7—C12	120.72 (15)
C2—C1—H1A	109.5	C8—C7—N2	118.75 (15)
C2—C1—H1B	109.5	C12—C7—N2	120.53 (15)
H1A—C1—H1B	109.5	C7—C8—C9	119.29 (18)
C2—C1—H1C	109.5	C7—C8—H8A	120.4

H1A—C1—H1C	109.5	C9—C8—H8A	120.4
H1B—C1—H1C	109.5	C10—C9—C8	120.18 (19)
O2—C2—C1	107.41 (16)	C10—C9—H9A	119.9
O2—C2—H2B	110.2	C8—C9—H9A	119.9
C1—C2—H2B	110.2	C11—C10—C9	120.30 (18)
O2—C2—H2C	110.2	C11—C10—H10A	119.9
C1—C2—H2C	110.2	C9—C10—H10A	119.9
H2B—C2—H2C	108.5	C10—C11—C12	120.33 (19)
O1—C3—O2	123.45 (14)	C10—C11—H11A	119.8
O1—C3—C4	125.29 (14)	C12—C11—H11A	119.8
O2—C3—C4	111.25 (13)	C11—C12—C7	119.16 (18)
C6—C4—C5	119.96 (13)	C11—C12—H12A	120.4
C6—C4—C3	122.00 (13)	C7—C12—H12A	120.4
C5—C4—C3	117.76 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.83 (2)	2.05 (2)	2.7210 (19)	137.9 (19)
N2—H2A···O1^{i}	0.83 (2)	2.54 (2)	3.1513 (19)	131.3 (18)
S1—H1···N1^{ii}	1.20 (2)	2.45 (2)	3.4560 (17)	140.1 (15)

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1