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ORIGINAL PAPER

Synthesis and Crystal Structure of 2-Amino-4-(4-hydroxyphenyl)-5-propylthiazole Ethanol Solvate 0.25 Hydrate

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Abstract 2-Amino-4-(4-hydroxyphenyl)-5-propylthiazole was synthesized by the reaction of α-bromo-1-(4-hydroxyphenyl)-1-pentone with thiourea. The crystal structure of its ethanol solvate 0.25 hydrate, $C_{12}H_{14}N_2OS\cdots C_2H_5OH\cdot\cdot$ $\cdot 0.25\cdot H_2O$, was determined by X-ray diffraction analysis. The crystal belongs to monoclinic system, space group C2/c with $a = 20.9046(10)$, $b = 10.1057(5)$, $c = 30.0017(15)$ Å, $\beta = 105.5850(10)°$, $Z = 8$, $M_r = 569.77$, $V = 6105.0(5)$ Å^3, $D_c = 1.240$ g/cm^3, $\mu = 0.214$ mm^{-1}, $F(000) = 2440$, the final $R = 0.0598$ and $wR = 0.1825$ for 5,911 observed reflections [$I > 2\sigma(I)$]. Compound (**1**) is composed by two non-coplanar ring systems of phenol and thiazole. The structure displays extensive O–H···N, N–H···O and O–H···O intermolecular hydrogen bonds.

Keywords Crystal structure · Synthesis · 2-Amino-4-(4-hydroxyphenyl)-5-propylthiazole

Introduction

The compounds containing thiazole ring are found to exhibit biological activities such as antiviral, antifungal and insecticidal activity [1]. A series of 5-(2-substituted-1,3-thiazol-5-yl)-2-hydroxy benzamides and their 2-alkoxy derivatives were synthesized and screened for their antifungal activities [2]. The 2,4-disubstituted thiazoles were prepared as anti-bacterial and anti-inflammatory agents [3], and a series of 4-phenyl thiazole derivatives were synthesized and tested their inhibitory effects on the interleukin-6 secretion stimulated by PTH in osteoblastic cells [4]. In this paper, the synthesis and characterization of 2-amino-4-(4-hydroxyphenyl)-5-propylthiazole (**1**) were reported. The chemistry to prepare compound (**1**) is depicted in Scheme 1.

Experimental

Melting points were measured on an RY-1 melting point apparatus and uncorrected. ^{1}H NMR spectra were recorded on a Varian Inova-400 with TMS as internal standard at 400 MHz with chemical shifts (δ) expressed in ppm. The IR spectra were obtained in a KBr disk using an AVATAR360FT spectrophotometer. The mass spectra were measured on a HP6890-5973.

Synthesis of Compound (1)

A mixture of α-bromo-1-(4-hydroxyphenyl)-1-pentone (0.01 mol) and thiourea (0.01 mol) in ethanol (50 mL) was refluxed for 3 h. Then the excess solvent was evaporated to give a yellow solid, which was recrystallized from ethanol to obtain compound (**2**) in 71.3% yield, m.p.: 165–166 °C.

^{1}H NMR (CD_3SOCD_3, ppm) δ: 0.87 (t, J = 7.6 Hz, 3H, CH_3), 1.54 (m, 2H, CH_2), 2.58 (t, J = 7.6 Hz, 2H, CH_2), 6.89 (d, J = 8.8 Hz, 2H, C_6H_4 3,5-H), 7.29 (d, J = 8.8 Hz, 2H, C_6H_4 2,6-H), 8.83 (br, 2H, NH_2), 9.92 (s, 1H, OH). IR

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Scheme 1 Synthetic route of compound (**1**)

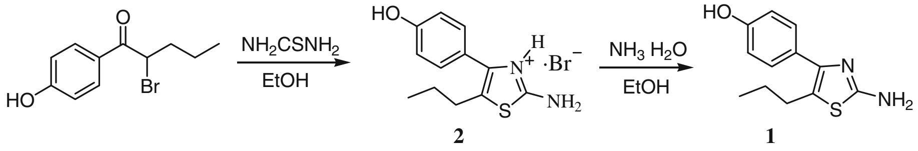

Table 1 Crystal data and structure refinement parameters

Compound	(**1**)
Empirical formula	$C_{28}H_{41}N_4O_{4.50}S_2$
Formula weight	569.77
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 20.9046(10) Å b = 10.1057(5) Å c = 30.0017(15) Å α = 90° β = 105.5850(10)° γ = 90°
Volume	6105.0(5) $Å^3$
Z	8
Density (calculated)	1.240 mg/m^3
Absorption coefficient	0.214 mm^{-1}
F(000)	2440
Crystal size (mm^3)	0.48 × 0.34 × 0.21
Theta range for data collection (°)	1.41–26.00
Index ranges	$-22 \le h \le 25$; $-8 \le k \le 12$; $-35 \le l \le 35$
Reflections collected	14220
Independent reflections	5911 [$R_{(int)}$ = 0.0303]
Data/restraints/parameters	5911/62/360
Goodness-of-fit on F^2	1.043
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0598 wR_2 = 0.1825
R indices (all data)	R_1 = 0.0854 wR_2 = 0.2084
Largest diff. peak and hole	0.900 and −0.629 e $Å^{-3}$

(KBr) ν/cm^{-1}: 3265 (NH_2), 1630 (C=N), 1583 (C=C), 1182 (C=S). And then compound (**2**) was converted to free-based aminothiazole by neutralization with ammonia in ethanol to give compound (**1**) in 62.6% yield, m.p.: 203–204 °C. ^{1}H NMR (CD_3SOCD_3, ppm) δ: 0.89 (t, J = 7.6 Hz, 3H, CH_3), 1.54 (m, 2H, CH_2), 2.62 (t, J = 7.6 Hz, 2H, CH_2), 6.78 (d, J = 8.4 Hz, 2H, C_6H_4 3,5-H), 7.07 (br, 2H, NH_2), 7.29 (d, J = 8.4 Hz, 2H, C_6H_4 2,6-H), 9.54 (s, 1H, OH). MS (m/z): 234 (M^+), 205, 163 (100%), 91, 80, 65, 41.

X-Ray Crystal Structure Determination

The crystals suitable for X-ray structure determination were obtained by slow evaporation of ethanol solution for

Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$)

Atom	x	y	z	U(eq)
S(1)	4276(1)	−125(1)	1236(1)	33(1)
S(2)	4290(1)	4932(1)	1248(1)	36(1)
C(1)	4751(1)	1311(3)	1326(1)	30(1)
C(2)	4396(1)	1256(3)	552(1)	25(1)
C(3)	4087(1)	133(3)	638(1)	28(1)
C(4)	4359(1)	1865(3)	98(1)	24(1)
C(5)	4400(1)	3250(3)	67(1)	26(1)
C(6)	4357(1)	3870(3)	−350(1)	26(1)
C(7)	4275(1)	3135(3)	−750(1)	26(1)
C(8)	4251(2)	1759(3)	−725(1)	30(1)
C(9)	4294(1)	1142(3)	−306(1)	29(1)
C(10)	3611(1)	−809(3)	330(1)	31(1)
C(11)	3906(2)	−2170(3)	284(1)	36(1)
C(12)	3383(2)	−3138(3)	20(1)	38(1)
C(13)	4371(2)	6324(3)	1596(1)	32(1)
C(14)	3273(1)	6202(3)	1315(1)	26(1)
C(15)	3431(1)	5127(3)	1094(1)	28(1)
C(16)	2612(1)	6786(3)	1266(1)	26(1)
C(17)	2549(1)	8154(3)	1307(1)	29(1)
C(18)	1940(1)	8750(3)	1257(1)	28(1)
C(19)	1366(1)	7992(3)	1161(1)	25(1)
C(20)	1416(2)	6629(3)	1125(1)	31(1)
C(21)	2030(1)	6041(3)	1179(1)	32(1)
C(22)	3015(2)	4163(3)	758(1)	31(1)
C(23)	2908(2)	2840(3)	969(1)	37(1)
C(24)	2509(2)	1880(3)	616(1)	45(1)
C(25)	6455(5)	2785(9)	2651(4)	193(4)
C(26)	6195(3)	4126(7)	2547(2)	130(3)
C(27)	3427(4)	9135(11)	2960(3)	172(4)
C(28)	3361(3)	8609(9)	2516(2)	128(3)
N(1)	4772(1)	1919(2)	946(1)	26(1)
N(2)	5071(1)	1706(3)	1756(1)	44(1)
N(3)	3814(1)	6876(2)	1603(1)	29(1)
N(4)	4983(1)	6730(3)	1855(1)	45(1)
O(1)	4222(1)	3703(2)	−1170(1)	31(1)
O(2)	772(1)	8619(2)	1103(1)	30(1)
O(3)	5531(2)	4125(3)	2276(1)	92(1)
O(4)	3943(1)	8380(3)	2388(1)	60(1)
O(5W)	5000	10002(4)	2500	49(1)

U(eq) is defined as one-third of the trace of the orthogonalized U_{ij} tensor

Table 3 Selected bond lengths (Å) and bond angles (°) for the title compound

Atoms	Length	Atoms	Length
S(1)–C(1)	1.739(3)	S(2)–C(13)	1.735(3)
S(1)–C(3)	1.751(3)	S(2)–C(15)	1.741(3)
C(1)–N(1)	1.307(4)	C(13)–N(3)	1.296(4)
C(1)–N(2)	1.344(4)	C(13)–N(4)	1.368(4)
C(2)–C(3)	1.363(4)	C(14)–C(15)	1.359(4)
C(2)–N(1)	1.400(4)	C(14)–N(3)	1.402(4)
C(2)–C(4)	1.480(4)	C(14)–C(16)	1.473(4)
C(3)–C(10)	1.502(4)	C(15)–C(22)	1.500(4)
C(4)–C(9)	1.391(4)	C(16)–C(21)	1.394(4)
C(4)–C(5)	1.406(4)	C(16)–C(17)	1.397(4)
C(5)–C(6)	1.379(4)	C(17)–C(18)	1.380(4)
C(5)–H(5)	0.9500	C(17)–H(17)	0.9500
C(6)–C(7)	1.382(4)	C(18)–C(19)	1.384(4)
C(7)–O(1)	1.363(3)	C(19)–O(2)	1.363(3)
C(7)–C(8)	1.395(4)	C(19)–C(20)	1.388(4)
C(8)–C(9)	1.383(4)	C(20)–C(21)	1.385(4)
C(10)–C(11)	1.531(4)	C(22)–C(23)	1.521(4)
C(10)–H(10A)	0.9900	C(22)–H(22A)	0.9900
C(11)–C(12)	1.521(4)	C(23)–C(24)	1.510(5)
C(11)–H(11A)	0.9900	C(23)–H(23A)	0.9900
C(12)–H(12A)	0.9800	C(24)–H(24A)	0.9800
C(25)–C(26)	1.513(11)	C(27)–C(28)	1.489(5)
C(25)–H(25A)	0.9800	C(27)–H(27A)	0.9800
C(26)–O(3)	1.404(6)	C(28)–O(4)	1.385(6)
C(26)–H(26A)	0.9900	C(28)–H(28A)	0.9900
N(2)–H(2A)	0.8800	N(4)–H(4A)	0.82(4)
O(1)–H(1)	0.8400	O(2)–H(2)	0.8400
O(3)–H(3)	0.8400	O(4)–H(4)	0.8400
O(5W)–H(5A)	0.73(4)		

Atoms	Angle	Atoms	Angle
C(1)–S(1)–C(3)	89.75(14)	C(13)–S(2)–C(15)	89.77(14)
N(1)–C(1)–N(2)	125.0(3)	N(3)–C(13)–N(4)	124.9(3)
N(1)–C(1)–S(1)	114.2(2)	N(3)–C(13)–S(2)	114.4(2)
N(2)–C(1)–S(1)	120.8(2)	N(4)–C(13)–S(2)	120.6(2)
C(3)–C(2)–N(1)	115.3(3)	C(15)–C(14)–N(3)	115.3(3)
C(3)–C(2)–C(4)	127.6(3)	C(15)–C(14)–C(16)	128.0(3)
N(1)–C(2)–C(4)	117.1(2)	N(3)–C(14)–C(16)	116.5(2)
C(2)–C(3)–C(10)	132.9(3)	C(14)–C(15)–C(22)	132.3(3)
C(2)–C(3)–S(1)	109.3(2)	C(14)–C(15)–S(2)	109.2(2)
C(10)–C(3)–S(1)	117.7(2)	C(22)–C(15)–S(2)	118.5(2)
C(9)–C(4)–C(5)	117.4(3)	C(21)–C(16)–C(17)	116.9(3)
C(9)–C(4)–C(2)	123.5(3)	C(21)–C(16)–C(14)	123.4(3)
C(5)–C(4)–C(2)	119.1(3)	C(17)–C(16)–C(14)	119.7(3)
C(6)–C(5)–C(4)	121.4(3)	C(18)–C(17)–C(16)	121.8(3)
C(6)–C(5)–H(5)	119.3	C(18)–C(17)–H(17)	119.1
C(4)–C(5)–H(5)	119.3	C(16)–C(17)–H(17)	119.1
C(5)–C(6)–C(7)	120.4(3)	C(17)–C(18)–C(19)	120.3(3)
C(5)–C(6)–H(6)	119.8	C(17)–C(18)–H(18)	119.9
C(7)–C(6)–H(6)	119.8	C(19)–C(18)–H(18)	119.9

Table 3 continued

Atoms	Angle	Atoms	Angle
O(1)–C(7)–C(6)	122.6(3)	O(2)–C(19)–C(18)	118.6(3)
O(1)–C(7)–C(8)	118.3(3)	O(2)–C(19)–C(20)	122.3(3)
C(6)–C(7)–C(8)	119.2(3)	C(18)–C(19)–C(20)	119.1(3)
C(9)–C(8)–C(7)	120.2(3)	C(21)–C(20)–C(19)	120.2(3)
C(9)–C(8)–H(8)	119.9	C(21)–C(20)–H(20)	119.9
C(7)–C(8)–H(8)	119.9	C(19)–C(20)–H(20)	119.9
C(8)–C(9)–C(4)	121.4(3)	C(20)–C(21)–C(16)	121.8(3)
C(8)–C(9)–H(9)	119.3	C(20)–C(21)–H(21)	119.1
C(4)–C(9)–H(9)	119.3	C(16)–C(21)–H(21)	119.1
C(3)–C(10)–C(11)	114.1(3)	C(15)–C(22)–C(23)	114.5(3)
C(3)–C(10)–H(10A)	108.7	C(15)–C(22)–H(22A)	108.6
C(11)–C(10)–H(10A)	108.7	C(23)–C(22)–H(22A)	108.6
H(10A)–C(10)–H(10B)	107.6	C(15)–C(22)–H(22B)	108.6
C(12)–C(11)–C(10)	111.9(3)	H(22A)–C(22)–H(22B)	107.6
C(12)–C(11)–H(11A)	109.2	C(24)–C(23)–C(22)	112.9(3)
C(10)–C(11)–H(11A)	109.2	C(24)–C(23)–H(23A)	109.0
C(12)–C(11)–H(11B)	109.2	C(22)–C(23)–H(23A)	109.0
H(11A)–C(11)–H(11B)	107.9	H(23A)–C(23)–H(23B)	107.8
C(11)–C(12)–H(12A)	109.5	C(23)–C(24)–H(24A)	109.5
H(12A)–C(12)–H(12B)	109.5	H(24A)–C(24)–H(24B)	109.5
H(12A)–C(12)–H(12C)	109.5	C(23)–C(24)–H(24C)	109.5
C(26)–C(25)–H(25A)	109.5	C(28)–C(27)–H(27A)	109.5
H(25A)–C(25)–H(25B)	109.5	H(27A)–C(27)–H(27B)	109.5
O(3)–C(26)–C(25)	111.5(6)	C(28)–C(27)–H(27C)	109.5
O(3)–C(26)–H(26A)	109.3	O(4)–C(28)–C(27)	115.5(6)
C(25)–C(26)–H(26A)	109.3	O(4)–C(28)–H(28A)	108.4
C(25)–C(26)–H(26B)	109.3	C(27)–C(28)–H(28A)	108.4
H(26A)–C(26)–H(26B)	108.0	H(28A)–C(28)–H(28B)	107.5
C(1)–N(1)–C(2)	111.6(2)	C(13)–N(3)–C(14)	111.3(3)
C(1)–N(2)–H(2A)	120.0	C(13)–N(4)–H(4A)	116(3)
H(2A)–N(2)–H(2B)	120.0	H(4A)–N(4)–H(4B)	113(4)
C(7)–O(1)–H(1)	109.5	C(19)–O(2)–H(2)	109.5
C(26)–O(3)–H(3)	109.5	C(28)–O(4)–H(4)	109.5

about 10 days at room temperature. A yellow single crystal of the title compound was selected and mounted in air onto thin glass fibers. X-ray intensity data were measured at 173(2) K on a Bruker AXS SMART 1000 CCD diffractometer equipped with a graphite-monochromatized Mo K_α (λ = 0.71073 Å) radiation. The crystal parameters and refinement data are listed in Table 1. Corrections for incident and diffracted beam absorption effects were applied using SADABS [5]. The structure was solved by direct methods with SHELXS-97 [6] and expanded by using *Fourier* difference techniques. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms bonded to N(4) and water molecule were located in a difference *Fourier* map and refined isotropically, the U_{iso}(H) values were set equal to $1.5U_{\mathrm{eq}}$(O) and $1.2U_{\mathrm{eq}}$(N), respectively. Other hydrogen atoms were refined in the riding-model approximation. The structure was refined by full-matrix least-squares techniques on F^2 with SHELXL-97 [7]. As a result, the atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms are given in Table 2. The selected bond lengths, bond angles and hydrogen bonds are listed in Tables 3 and 4, respectively.

Results and Discussion

The ^{1}H NMR for the product is in good agreement with the structure of (**1**). The spectrum showed a singlet at δ 9.54 due to the OH group on benzene ring. A broad singlet at δ

Table 4 Hydrogen bonds lengths (Å) and bond angles (°) for the title compound

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	∠(DHA)
N(2)–H(2A)···O(3)	0.88	2.21	2.918(5)	137.7
N(2)–H(2B)···O(5W)^{i}	0.88	1.98	2.856(4)	172.0
N(4)–H(4A)···O(4)^{ii}	0.82(4)	2.65(4)	3.199(4)	125(4)
N(4)–H(4B)···O(1)^{iii}	0.82(4)	2.21(4)	3.008(4)	163(4)
O(1)–H(1)···O(2)^{iv}	0.84	1.87	2.714(3)	177.7
O(2)–H(2)···N(1)^{vi}	0.84	1.81	2.648(3)	176.6
O(3)–H(3)···N(4)	0.84	2.19	3.001(5)	161.7
O(4)–H(4)···N(3)	0.84	1.92	2.755(4)	170.0
O(5W)–H(5A)···O(4)	0.73(4)	1.97(4)	2.698(4)	176(6)

Symmetry codes: (i) x, $y - 1$, z; (ii) $-x + 1$, y, $-z + 1/2$; (iii) $-x + 1$, $-y + 1$, $-z$; (iv) $-x + 1/2$, $-y + 3/2$, $-z$; (vi) $x - 1/2$, $y + 1/2$, z

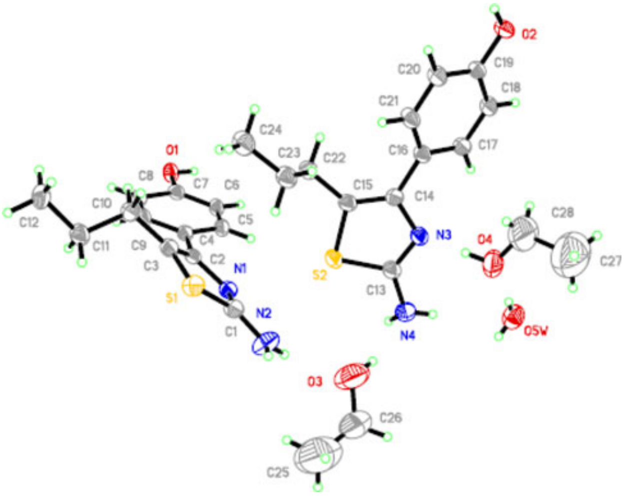

Fig. 1 The molecular structure of (**1**), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. The water molecule lies on a crystallographic two-fold axis

7.07 corresponding to NH_2 proton. Two doublets in the range of δ 6.78 (J = 8.4 Hz) and δ 7.29 (J = 8.4 Hz) are due to the aromatic protons on the benzene ring. Propyl group presenting as the side chain showed two triplets at δ 0.89 (J = 7.6 Hz) and δ 2.62 (J = 7.6 Hz). Mass spectrum of (**1**) showed the molecular ion peak at *m*/*z* 234, which is in accordance with its base's molecular formula $C_{12}H_{14}N_2OS$, and a base peak at *m*/*z* 163(100%) due to fragmentation of M^+-$COCH_2CH_2CH_3$. IR spectrum of (**1**) showed absorption bands at 3265, 1630, 1583 and 1182 cm^{-1} due to NH_2, C=N, C=C, C=S groups, respectively. The melting point of compound (**1**) (base, m.p.: 203–204 °C.) is higher than its hydrobromide [compound (**2**), m.p.: 165–166 °C], which is quite different from the usual instance [8–10].

In the crystal structure of the title compound (Fig. 1), the molecular contains two independent molecules of compound **1**, two molecules of ethanol and a half molecule of water, which is located on a two-fold rotation axis. For the existence of solvent ethanol in the crystal structure, C–C distances and anisotropic vibration factors in the ethanol were restrained in the process of refinement. Finally, The distance of highest peak (0.88 e Å^{-3}) from H(26B) was 0.55 Å. Compound (**1**) is composed by two ring systems of phenol and thiazole. The dihedral angle between the planes of the benzene and thiazole ring is 31.5(1)° in the molecular including S(1), and The dihedral angle between the planes of the benzene and thiazole ring is 31.1(2)° in the molecular including S(2).

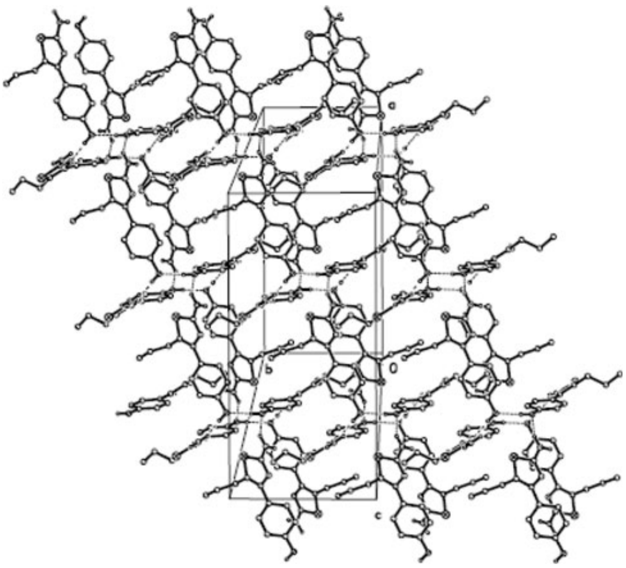

Fig. 2 The hydrogen bonding diagram for the title compound

The arrangement of the molecules in the unit cell is shown in Fig. 2. The crystal is stabilized by the intermolecular hydrogen bonds, which are N(4)–H(4B)···O(1)^{iii} between the amino group of the thiazole ring and the oxygen atom of a neighbouring molecule and O(2)–H(2)···N(1)^{vi} between the hydroxyl group of the phenyl ring and the nitrogen atom of the thiazole ring. In addition, the hydroxyl group of the phenyl ring also acts as a hydrogen bond donor to another molecule's oxygen atom, forming the intermolecular hydrogen bond O(1)–H(1)···O(2)^{iv}.

As it is shown in Fig. 2, the structure of the title compound, $C_{12}H_{14}N_2OS \cdot EtOH \cdot 0.25H_2O$, comprises molecules of the thiazole in an extensive hydrogen-bonding network with ethanol molecules and water molecules (Table 4), such as an intermolecular hydrogen bond O(4)–H(4)···N(3) between the hydroxyl group of the ethanol molecule and the nitrogen atom of the thiazole ring. The amino group of the thiazole and the hydroxyl group of ethanol molecule also form intermolecular hydrogen bonds of type N(4)–H(4A)···O(4)^{ii} and O(3)–H(3)···N(4). Moreover, the formation of hydrogens bond between the crystallization water molecule, the amino group of the thiazole and ethanol molecule were also observed [e.g. N(2)–H(2B)···O(5W)^{i}, O(5W)–H(5A)···O(4)].

Supplementary Material

CCDC-291112 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033; email:deposit@ccdc.cam.ac.uk].

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