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Full Length Research Paper

Co-crystal structure of mixed molecules

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Based on the fundamentals of organic chemistry, the approach leads to the corresponding amides in good yields which were the reaction of esters with primary amines. In our laboratory, there was no reaction when we tried to react methyl 2-(2-oxo-2H-chromen-7-yloxy)acetate with 2-(2-aminophenyl) benzothiazole). Although the spectral data was so enough for validation of the structure of the product but in the thin layer chromatography (TLC) test, there still appear two spots for the reactant (no reaction); so only X-ray single crystallography will solve this problem and identify the structure of this shiny light brown crystal. Co-crystal structure was synthesized in methanolic solution from methyl 2-(2-oxo-2H-chromen-7-yloxy)acetate and 2-(2-aminophenyl)benzothiazole). Single X-ray crystallography was studied and it was found that there was no hydrogen bonding between the molecules, moreover, the co-crystal is very stable.

Key words: 2-(2-aminophenyl)benzothiazole), co-crystal, coumarins, methanol, single-crystal X-ray.

INTRODUCTION

During the last twenty years, the study of the biological activities of coumarin derivatives has been the aim of many researchers (Shih et al., 2007; Li et al., 2007; Nofal et al., 2002; Al-Amiery et al., 2011; Kadhum et al., 2011a, 2011b). Although most of the existing natural coumarins have been isolated from higher plants, some of them have been discovered in microorganisms, for example, aminocoumarin antibiotics: novobiocin, coumermycin A1 and chlorobiocin (produced by the actinomycete *Streptomyces niveus*) (Završniket al., 2011). Synthetic coumarin derivatives have been obtained by chemical modification of the coumarin ring. Coumarins and their derivatives have attracted considerable attention due to their extensively biological activities such as antibacterial, antifungal, antiviral, anti-tubercular, anti-malarial, anticoagulant, anti-inflammatory, anticancer, antioxidant properties and so on. Numerous efforts including the separation and purification of naturally occurring coumarins from a variety of plants as well as artificial synthesis of coumarin compounds with novel structures and properties have been focusing on the research and development of coumarins as potential drugs. So far, some coumarins, for example, warfarin, acenocoumarol, armillarisin A, hymecromone and carbochromen have been approved for therapeutic purposes in clinic. More importantly, an increasing number of coumarin compounds have displayed great potency in the treatment of various types of diseases (Shi and Zhou, 2011). Structure activity relationships of coumarin derivatives have revealed that the presence of substituted amino derivatives is an essential feature of their pharmacological action. Based on these findings, we try to describe the synthesis of some compounds featuring different heterocyclic rings fused onto the coumarin moiety with the aim of obtaining more potent pharmacologically active compounds, but no reaction has been done. The structures of methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy)acetate and 2-(2-aminophenyl) benzothiazole) are shown in Figure 1.

EXPERIMENTAL

The chemicals used during synthesis were supplied by Sigma-Aldrich. Purity of the compounds was checked on thin layer chromatography (TLC) plates (Silica gel G) in the solvent system benzene: ethyl acetate: methanol (40:30:30, v/v/v) and toluene:

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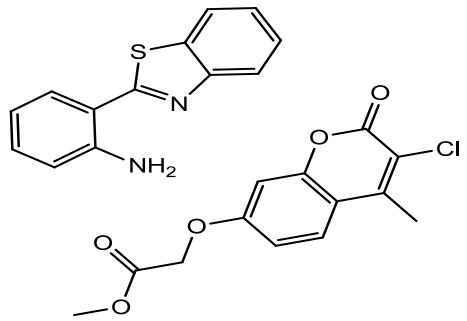

Figure 1. The structure of the Co-crystal [Methyl2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy)acetate and 2-(2-aminopheny l)benzothiazole)].

acetone (75:25, v/v). The spots were located under ultraviolet (UV) light 254 and 365 nm.

General procedure for synthesis of co-crystal

Mixture of 0.05 mol of 2-(2-aminophenyl)benzothiazole and 0.05 mol of methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy) acetate in methanol were refluxed for 2 h, then suitable co-crystal appeared by evaporation of methanol solution.

Crystal structure determination

Diffraction data was collected on a Bruker APEX CCD area-detector diffractometer at 298(2) K on a crystal size 0.50 x 0.33 x 0.26 mm with Mo Kα radiation (Wavelength 0.71073 ^{o}A. Empirical formula C26 H21 Cl N2 O5 S, formula weight 508.96, unit cell dimensions a = 12.611(2) A alpha = 90°, b = 7.3544(13) A beta = 99.031(4)°, c = 25.980(5), A gamma = 90 deg. volume 2379.7(7) A^3 Z, calculated density 4, 1.421 Mg/m^3. Absorption coefficient 0.290 mm^-1. F(000) 1056. Theta range for data collection 1.59 to 25.50°, limiting indices -15<=h<=15, -8<=k<=8, -31<=l<=23. Reflections collected / unique 13480 / 4416 [R(int) = 0.0263]. Completeness to theta = 25.50 99.8%. Maximum and minimum transmission 0.9285 and 0.8687. Refinement method full-matrix least-squares on F^2. Data / restraints / parameters 4416 / 0 / 321. Goodness-of-fit on F^2 1.033. Final R indices [I>2sigma(I)] R1 = 0.0520, wR2 = 0.1314. R indices (all data) R1 = 0.0679, wR2 = 0.1435. Largest difference peak and hole 0.524 and -0.411 e.A^-3.

RESULTS AND DISCUSSION

The interest in co-crystals driven largely by new opportunities in the design and construction of solid-state materials (Jones, 1997) has also brought about an equally strong interest to discover new and efficient methods of co-crystal synthesis. Specifically, in addition to the traditional means of constructing co-crystals by co-crystallization from solution, several research groups have described the construction of two component or binary co-crystals by grinding together the two co-crystal components (Etter et al., 1993). It is too difficult to synthesize co-crystal. Many researches try to build a co-

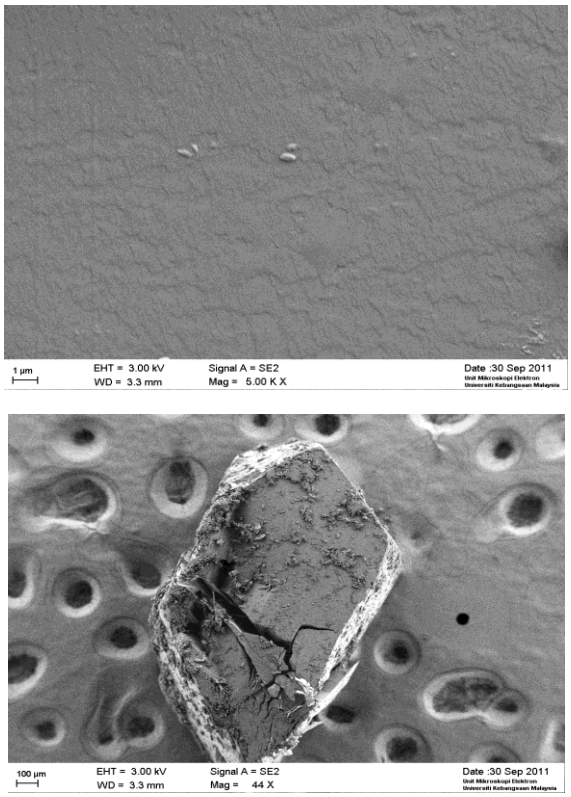

Figure 2. FESEM for the Co-crystal [Methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy)acetate and 2-(2-aminophenyl)benzothiazole)].

crystal but the results were not encouraged; all attempts to obtain co-crystals of theophylline, D/Ltartaric acid from solution failed (the outcome was theophylline). It was not possible to elucidate the structure of the co-crystal using single crystal X-ray diffraction (Tomislav and William 2007; Andrei and Dimitrii, 2003; Juliana et al., 2011; Nur et al., 2010; Bohari and Nur, 2011; En-Jun et al., 2007; Sekhon 2009) Figure 2a shows an SEM image of a growing co-crystal that has been synthesized from crystal grown from solution of methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy)acetate and 2-(2-aminophenyl) benzothiazole), at 100 µm. Figure 2b shows the SEM image of surface morphologies of a co-crystal at 1 µm. From the crystal structure (Figure 3), it was found that there were two molecules in the crystal structure, first one was methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy)acetate and the second 2-(2-aminophenyl) benzothiazole). The crystal structure co-crystal is shown in Figure 3. The crystal data and experimental details are shown in Table 1. The bond lengths and bond angles of co-crystal are shown in Table 2. Anisotropic displacement parameters and isotropic displacement parameters are listed in Tables 4 and 5. The single crystal of co-crystal suitable for X-ray diffraction analyses was obtained from methanol solutions and the structure was examined by a single-crystal X-ray diffraction analysis. As shown in

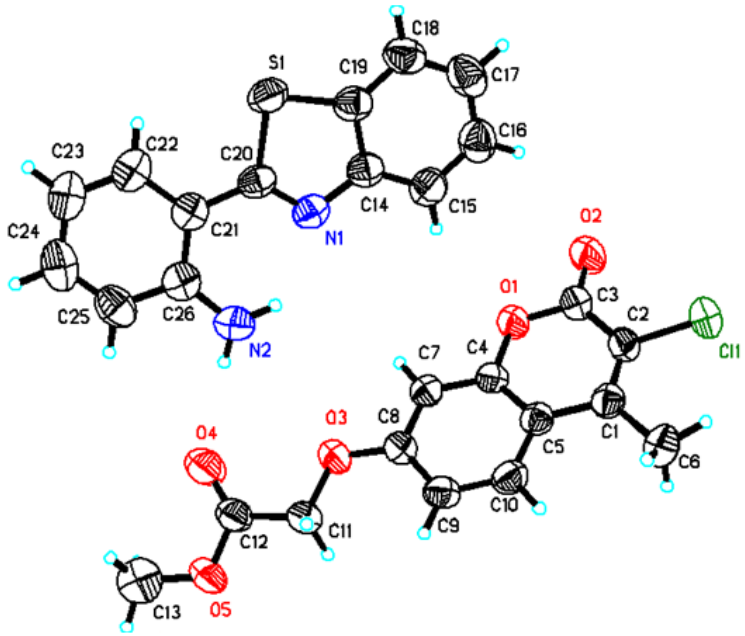

Figure 3. The crystal structure for co-crystal [Methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy)acetate and 2-(2-aminophenyl) benzothiazole)].

Table 1. Crystal data and structure refinement for Co-crystal baru441m.

Crystal data	Structure refinement
Identification code	baru441m
Empirical formula	C_{26} H_{21} Cl N_2 O_5 S
Formula weight	508.96
Temperature	298(2) K
Wavelength	0.71073 A
Unit cell dimensions	a = 12.611(2) A alpha = 90° b = 7.3544(13) A beta = 9.031(4) ° c = 25.980(5) A gamma = 90°
Volume	2379.7(7) A^3
Z, Calculated density	4, 1.421 Mg/m^3
Absorption coefficient	0.290 mm^{-1}
F(000)	1056
Crystal size	0.50 x 0.33 x 0.26 mm
Theta range for data collection	1.59 to 25.50°
Limiting indices	-15<=h<=15, -8<=k<=8, -31<=l<=23
Reflections collected / unique	13480 / 4416 [R(int) = 0.0263]
Completeness to theta	25.50 99.8%
Max. and min. transmission	0.9285 and 0.8687
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4416 / 0 / 321
Goodness-of-fit on	F^2 = 1.033
Final R indices	[I>2sigma(I)] R1 = 0.0520, wR2 = 0.1314
R indices (all data)	R1 = 0.0679, wR2 = 0.1435
Largest diff. peak and hole	0.524 and -0.411 $e.A^{-3}$

Table 2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($A^2 \times 10^3$) for baru441m. *U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.*

Atom	X	Y	Z	U(eq)
S(1)	2810(1)	7553(1)	2187(1)	61(1)
O(1)	7347(1)	3552(3)	1460(1)	57(1)
O(2)	6800(2)	3195(4)	624(1)	78(1)
O(3)	8198(1)	4103(3)	3260(1)	62(1)
O(4)	7442(2)	3735(4)	4160(1)	82(1)
O(5)	9100(2)	3946(3)	4599(1)	68(1)
N(1)	4501(2)	5511(3)	2361(1)	53(1)
N(2)	5626(2)	5121(4)	3347(1)	68(1)
C(1)	9270(2)	5128(3)	1265(1)	48(1)
C(2)	8519(2)	4522(4)	877(1)	52(1)
C(3)	7508(2)	3722(4)	957(1)	55(1)
C(4)	8091(2)	4130(3)	1877(1)	45(1)
C(5)	9051(2)	4932(3)	1795(1)	44(1)
C(6)	10305(2)	5958(4)	1166(1)	63(1)
C(7)	7819(2)	3875(4)	2361(1)	51(1)
C(8)	8534(2)	4411(4)	2793(1)	49(1)
C(9)	9505(2)	5209(4)	2732(1)	53(1)
C(10)	9748(2)	5465(4)	2241(1)	51(1)
C(11)	8922(2)	4500(4)	3719(1)	54(1)
C(12)	8383(2)	4002(4)	4171(1)	55(1)
C(13)	8694(3)	3587(5)	5079(1)	87(1)
C(14)	4274(2)	5761(4)	1828(1)	52(1)
C(15)	4861(2)	5030(4)	1464(1)	63(1)
C(16)	4541(3)	5406(4)	946(1)	69(1)
C(17)	3656(3)	6486(5)	780(1)	70(1)
C(18)	3062(2)	7215(4)	1129(1)	65(1)
C(19)	3373(2)	6849(4)	1655(1)	54(1)
C(20)	3814(2)	6354(3)	2601(1)	50(1)
C(21)	3825(2)	6377(3)	3164(1)	52(1)
C(22)	2929(2)	7040(4)	3366(1)	65(1)
C(23)	2896(3)	7106(5)	3888(1)	75(1)
C(24)	3781(3)	6514(5)	4230(1)	77(1)
C(25)	4668(3)	5851(4)	4046(1)	68(1)
C(26)	4722(2)	5760(3)	3512(1)	55(1)
Cl(1)	8685(1)	4652(1)	230(1)	76(1)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Figure 3, there are no hydrogen bonds between (Table 7) methyl 2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy) acetate and 2-(2-aminophenyl) benzothiazole).

2-(2-aminophenyl) benzothiazol

The benzothiazole group is not coplanar with the aryl ring, this is attributed to steric interaction of aryl group and amino group with the nitrogen of benzothiazole, in addition, the bond length of C(20)-C(21) is1.461 and the bonds angle of C(22)-C(21)-C(20) is 119.7 and C(26)-C(21)-C(20) is 121.4 (Table 3). Moreover, the torsion

Table 3. Bond lengths [A] and Bond angles [deg] for baru441 m.

Bonds length	Angle
S(1)-C(19)	1.729(3)
S(1)-C(20)	1.763(3)
O(1)-C(3)	1.360(3)
O(1)-C(4)	1.383(3)
O(2)-C(3)	1.206(3)
O(3)-C(8)	1.364(3)
O(3)-C(11)	1.414(3)
O(4)-C(12)	1.199(3)
O(5)-C(12)	1.318(3)
O(5)-C(13)	1.444(4)
N(1)-C(20)	1.302(3)
N(1)-C(14)	1.382(3)
N(2)-C(26)	1.363(4)
N(2)-H(2A)	0.82(3)
N(2)-H(2B)	0.91(3)
C(1)-C(2)	1.346(4)
C(1)-C(5)	1.452(3)
C(1)-C(6)	1.499(4)
C(2)-C(3)	1.449(4)
C(2)-Cl(1)	1.730(2)
C(4)-C(7)	1.368(3)
C(4)-C(5)	1.393(3)
C(5)-C(10)	1.396(3)
C(6)-H(6A)	0.9600
C(6)-H(6B)	0.9600
C(6)-H(6C)	0.9600
C(7)-C(8)	1.382(3)
C(7)-H(7)	0.9300
C(8)-C(9)	1.390(4)
C(9)-C(10)	1.372(4)
C(9)-H(9)	0.9300
C(9)-H(9)	0.9300
C(10)-H(10)	0.9300
C(11)-C(12)	1.493(4)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(13)-H(13A)	0.9600
C(13)-H(13B)	0.9600
C(13)-H(13C)	0.9600
C(15)-C(16)	1.371(4)
C(15)-C(14)	1.396(4)
C(15)-H(15A)	0.9300
C(16)-C(17)	1.383(5)
C(16)-H(16A)	0.9300
C(17)-C(18)	1.374(4)
C(17)-H(17A)	0.9300
C(18)-C(19)	1.389(4)
C(18)-H(18A)	0.9300
C(19)-C(14)	1.404(4)
C(20)-C(21)	1.461(4)

Table 3. Contd.

C(21)-C(22)	1.407(4)
C(21)-C(26)	1.408(4)
C(22)-C(23)	1.364(4)
C(22)-H(22A)	0.9300
C(23)-C(24)	1.383(5)
C(23)-H(23A)	0.9300
C(24)-C(25)	1.373(5)
C(24)-H(24A)	0.9300
C(25)-C(26)	1.401(4)
C(25)-H(25)	0.9300
C(19)-S(1)-C(20)	89.56(13)
C(3)-O(1)-C(4)	122.55(19)
C(8)-O(3)-C(11)	117.8(2)
C(12)-O(5)-C(13)	116.4(2)
C(20)-N(1)-C(14)	111.6(2)
C(26)-N(2)-H(2A)	115(2)
C(26)-N(2)-H(2B)	118(2)
H(2A)-N(2)-H(2B)	127(3)
C(2)-C(1)-C(5)	117.5(2)
C(2)-C(1)-C(6)	122.3(2)
C(5)-C(1)-C(6)	120.2(2)
C(1)-C(2)-C(3)	123.9(2)
C(1)-C(2)-Cl(1)	122.1(2)
C(3)-C(2)-Cl(1)	113.91(19)
O(2)-C(3)-O(1)	117.0(2)
O(2)-C(3)-C(2)	126.7(2)
O(1)-C(3)-C(2)	116.3(2)
C(7)-C(4)-O(1)	116.1(2)
C(7)-C(4)-C(5)	123.2(2)
O(1)-C(4)-C(5)	120.7(2)
C(4)-C(5)-C(10)	116.2(2)
C(4)-C(5)-C(1)	119.0(2)
C(10)-C(5)-C(1)	124.7(2)
C(1)-C(6)-H(6A)	109.5
C(1)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
C(1)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
C(4)-C(7)-C(8)	118.9(2)
C(4)-C(7)-H(7)	120.6
C(8)-C(7)-H(7)	120.6
O(3)-C(8)-C(7)	114.9(2)
O(3)-C(8)-C(9)	125.0(2)
C(7)-C(8)-C(9)	120.1(2)
C(10)-C(9)-C(8)	119.6(2)
C(10)-C(9)-H(9)	120.2
C(8)-C(9)-H(9)	120.2
C(9)-C(10)-C(5)	122.0(2)
C(9)-C(10)-H(10)	119.0
C(5)-C(10)-H(10)	119.0
O(3)-C(11)-C(12)	107.5(2)

Table 3. Cont'd.

O(3)-C(11)-H(11A)	110.2
C(12)-C(11)-H(11A)	110.2
O(3)-C(11)-H(11B)	110.2
C(12)-C(11)-H(11B)	110.2
H(11A)-C(11)-H(11B)	108.5
O(4)-C(12)-O(5)	124.1(3)
O(4)-C(12)-C(11)	126.3(2)
O(5)-C(12)-C(11)	109.6(2)
O(5)-C(13)-H(13A)	109.5
O(5)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
O(5)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(16)-C(15)-C(14)	118.8(3)
C(16)-C(15)-H(15A)	120.6
C(14)-C(15)-H(15A)	120.6
C(15)-C(16)-C(17)	121.4(3)
C(15)-C(16)-H(16A)	119.3
C(17)-C(16)-H(16A)	119.3
C(18)-C(17)-C(16)	121.0(3)
C(18)-C(17)-H(17A)	119.5
C(16)-C(17)-H(17A)	119.5
C(17)-C(18)-C(19)	118.4(3)
C(17)-C(18)-H(18A)	120.8
C(19)-C(18)-H(18A)	120.8
C(18)-C(19)-C(14)	121.0(3)
C(18)-C(19)-S(1)	129.9(2)
C(14)-C(19)-S(1)	109.19(19)
N(1)-C(20)-C(21)	125.6(2)
N(1)-C(20)-S(1)	114.5(2)
C(21)-C(20)-S(1)	119.91(19)
N(1)-C(14)-C(15)	125.4(2)
N(1)-C(14)-C(19)	115.2(2)
C(15)-C(14)-C(19)	119.4(3)
C(22)-C(21)-C(26)	118.9(3)
C(22)-C(21)-C(20)	119.7(2)
C(26)-C(21)-C(20)	121.4(2)
C(23)-C(22)-C(21)	122.3(3)
C(23)-C(22)-H(22A)	118.8
C(21)-C(22)-H(22A)	118.8
C(22)-C(23)-C(24)	118.7(3)
C(22)-C(23)-H(23A)	120.7
C(24)-C(23)-H(23A)	120.7
C(25)-C(24)-C(23)	120.6(3)
C(25)-C(24)-H(24A)	119.7
C(23)-C(24)-H(24A)	119.7
C(24)-C(25)-C(26)	122.0(3)
C(24)-C(25)-H(25)	119.0
C(26)-C(25)-H(25)	119.0
N(2)-C(26)-C(25)	120.1(3)
N(2)-C(26)-C(21)	122.3(3)
C(25)-C(26)-C(21)	117.5(3)

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for baru441 m.

Atom	U11	U22	U33	U23	U13	U12
S(1)	47(1)	65(1)	69(1)	4(1)	8(1)	10(1)
O(1)	47(1)	81(1)	42(1)	-2(1)	5(1)	-10(1)
O(2)	65(1)	121(2)	46(1)	-4(1)	0(1)	-16(1)
O(3)	52(1)	95(2)	39(1)	0(1)	4(1)	-8(1)
O(4)	64(1)	126(2)	53(1)	17(1)	1(1)	-21(1)
O(5)	64(1)	96(2)	40(1)	10(1)	-2(1)	-4(1)
N(1)	48(1)	53(1)	60(1)	2(1)	9(1)	5(1)
N(2)	57(2)	83(2)	61(2)	7(1)	0(1)	8(1)
C(1)	48(1)	44(1)	52(1)	6(1)	13(1)	8(1)
C(2)	54(1)	60(2)	43(1)	11(1)	12(1)	9(1)
C(3)	54(1)	69(2)	43(1)	1(1)	9(1)	4(1)
C(4)	41(1)	51(1)	42(1)	-1(1)	3(1)	3(1)
C(5)	43(1)	41(1)	48(1)	2(1)	9(1)	6(1)
C(6)	60(2)	66(2)	67(2)	3(1)	22(1)	-7(1)
C(7)	41(1)	63(2)	48(1)	1(1)	8(1)	-5(1)
C(8)	48(1)	56(2)	43(1)	1(1)	6(1)	3(1)
C(9)	44(1)	62(2)	50(1)	-5(1)	0(1)	0(1)
C(10)	42(1)	56(2)	57(2)	1(1)	8(1)	-3(1)
C(11)	49(1)	68(2)	44(1)	1(1)	0(1)	5(1)
C(12)	54(2)	63(2)	46(1)	6(1)	0(1)	-1(1)
C(15)	57(2)	67(2)	66(2)	-5(1)	13(1)	0(1)
C(16)	72(2)	76(2)	61(2)	-11(2)	15(2)	-12(2)
C(17)	72(2)	82(2)	54(2)	1(2)	1(2)	-24(2)
C(18)	56(2)	70(2)	66(2)	9(2)	-2(1)	-8(1)
C(19)	44(1)	54(2)	61(2)	1(1)	4(1)	-8(1)
C(20)	41(1)	45(1)	62(2)	2(1)	6(1)	-4(1)
C(14)	47(1)	50(2)	58(2)	0(1)	6(1)	-5(1)
C(21)	53(1)	44(1)	59(2)	-1(1)	11(1)	-6(1)
C(22)	64(2)	60(2)	76(2)	-2(2)	22(2)	1(1)
C(23)	84(2)	73(2)	74(2)	-6(2)	32(2)	1(2)
C(24)	103(3)	69(2)	62(2)	-11(2)	27(2)	-15(2)
C(25)	82(2)	61(2)	57(2)	0(1)	3(2)	-12(2)
C(26)	59(2)	45(1)	60(2)	-1(1)	9(1)	-9(1)
Cl(1)	74(1)	110(1)	46(1)	12(1)	17(1)	3(1)

The anisotropic displacement factor exponent takes the form:-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12].

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for baru441 m.

Atom	x	y	z	U(eq)
H(6A)	10881	5115	1266	95
H(6B)	10439	7054	1367	95
H(6C)	10256	6237	802	95
H(7)	7165	3349	2399	61
H(9)	9987	5567	3022	63
H(10)	10397	6010	2203	62
H(11A)	9102	5783	3731	65
H(11B)	9578	3806	3727	65
H(13A)	8226	4559	5147	131
H(13B)	9283	3501	5361	131
H(13C)	8302	2463	5048	131
H(15A)	5457	4302	1571	76
H(16A)	4928	4924	701	83
H(17A)	3461	6721	426	84
H(18A)	2465	7937	1015	78
H(22A)	2337	7449	3136	78
H(23A)	2291	7541	4012	90
H(24A)	3775	6565	4587	92
H(25)	5251	5450	4283	81
H(2A)	6090(20)	4750(40)	3579(12)	64(9)
H(2B)	5610(30)	4970(40)	2999(13)	77(10)

Table 6. Torsion angles (deg) for baru441 m.

Torsion angle	
C(5)-C(1)-C(2)-C(3)	1.1(4)
C(6)-C(1)-C(2)-C(3)	-179.6(3)
C(5)-C(1)-C(2)-Cl(1)	-179.32(17)
C(6)-C(1)-C(2)-Cl(1)	0.0(4)
C(4)-O(1)-C(3)-O(2)	-179.0(3)
C(4)-O(1)-C(3)-C(2)	1.0(4)
C(1)-C(2)-C(3)-O(2)	178.3(3)
Cl(1)-C(2)-C(3)-O(2)	-1.4(4)
C(1)-C(2)-C(3)-O(1)	-1.7(4)
Cl(1)-C(2)-C(3)-O(1)	178.64(19)
C(3)-O(1)-C(4)-C(7)	179.9(2)
C(3)-O(1)-C(4)-C(5)	0.3(4)
O(1)-C(4)-C(5)-C(10)	-179.9(2)
C(7)-C(4)-C(5)-C(1)	179.5(2)
O(1)-C(4)-C(5)-C(1)	-0.9(3)
C(2)-C(1)-C(5)-C(4)	0.3(3)
C(6)-C(1)-C(5)-C(4)	-179.0(2)
C(2)-C(1)-C(5)-C(10)	179.1(2)
C(6)-C(1)-C(5)-C(10)	-0.2(4)
O(1)-C(4)-C(7)-C(8)	179.5(2)
C(5)-C(4)-C(7)-C(8)	-0.9(4)
C(11)-O(3)-C(8)-C(7)	175.6(2)
C(11)-O(3)-C(8)-C(9)	-4.9(4)
C(4)-C(7)-C(8)-O(3)	-179.9(2)

angles for C(22)-C(21)-C(26)-N(2) is -179.1 and C(20)-C(21)-C(26)-N(2) is 0.4,b (Table 6).

2-(3-chloro-4-methyl-2-oxo-2H-chromen-7-yloxy) acetate (coumarin derivative)

From Figure 3, it is clear that coumarin molecule is planer, but the steric interaction of the extension of acetyl group is at O(3). The bond lengths of O(3)-C(8) is 1.364 and O(3)-C(11) is 1.414, and the bond angles for O(3)-C(8)-C(7) is 114.9, O(3)-C(8)-C(9) 125.0 and for C(8)-

Table 6. Contd.

C(4)-C(7)-C(8)-C(9)	0.6(4)
O(3)-C(8)-C(9)-C(10)	-179.3(2)
C(7)-C(8)-C(9)-C(10)	0.2(4)
C(8)-C(9)-C(10)-C(5)	-0.6(4)
C(4)-C(5)-C(10)-C(9)	0.3(4)
C(1)-C(5)-C(10)-C(9)	-178.6(2)
C(8)-O(3)-C(11)-C(12)	-178.4(2)
C(13)-O(5)-C(12)-O(4)	-1.8(5)
C(13)-O(5)-C(12)-C(11)	176.4(3)
O(3)-C(11)-C(12)-O(4)	-16.3(4)
O(3)-C(11)-C(12)-O(5)	165.5(2)
C(14)-C(15)-C(16)-C(17)	0.0(4)
C(15)-C(16)-C(17)-C(18)	-0.3(5)
C(16)-C(17)-C(18)-C(19)	0.3(4)
C(17)-C(18)-C(19)-C(14)	0.0(4)
C(17)-C(18)-C(19)-S(1)	179.8(2)
C(20)-S(1)-C(19)-C(18)	-179.7(3)
C(20)-S(1)-C(19)-C(14)	0.1(2)
C(14)-N(1)-C(20)-C(21)	-179.7(2)
C(14)-N(1)-C(20)-S(1)	-0.1(3)
C(19)-S(1)-C(20)-N(1)	0.0(2)
C(19)-S(1)-C(20)-C(21)	179.6(2)
C(20)-N(1)-C(14)-C(15)	-179.8(2)
C(20)-N(1)-C(14)-C(19)	0.2(3)
C(16)-C(15)-C(14)-N(1)	-179.6(3)
C(16)-C(15)-C(14)-N(1)	-179.6(3)
C(16)-C(15)-C(14)-C(19)	0.3(4)
C(18)-C(19)-C(14)-N(1)	179.6(2)
S(1)-C(19)-C(14)-N(1)	-0.2(3)
C(18)-C(19)-C(14)-C(15)	-0.3(4)
S(1)-C(19)-C(14)-C(15)	179.9(2)
N(1)-C(20)-C(21)-C(22)	-167.9(2)
S(1)-C(20)-C(21)-C(22)	12.6(3)
N(1)-C(20)-C(21)-C(26)	12.6(4)
S(1)-C(20)-C(21)-C(26)	-166.90(19)
C(26)-C(21)-C(22)-C(23)	-0.1(4)
C(20)-C(21)-C(22)-C(23)	-179.6(3)
C(21)-C(22)-C(23)-C(24)	0.6(5)
C(22)-C(23)-C(24)-C(25)	-0.8(5)
C(23)-C(24)-C(25)-C(26)	0.6(5)
C(24)-C(25)-C(26)-N(2)	178.9(3)
C(24)-C(25)-C(26)-C(21)	0.0(4)
C(22)-C(21)-C(26)-N(2)	-179.1(3)
C(20)-C(21)-C(26)-N(2)	0.4(4)
C(22)-C(21)-C(26)-C(25)	-0.2(4)
C(20)-C(21)-C(26)-C(25)	179.3(2)

Table 7. Hydrogen bonds for baru441 m (A and deg).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

O(3)-C(11) is 117.8(2) (Table 3). Moreover, the torsion angles for C(8)-O(3)-C(11)-C(12) is -178.4, C(11)-O(3)-C(8)-C(7) is 175.6 and C(11)-O(3)-C(8)-C(9) is -4.9 (Table 6).

CONCLUSIONS

In this study, coumarin derivative has been unsuccessfully synthesized. Structure of stale shiny light brown crystal was determined by X-ray single crystallography and it was found that there was no hydrogen bonding between the molecules, methyl 2-(2-oxo-2H-chromen-7-yloxy) acetate and 2-(2-aminophenyl)benzothiazole).

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