

## Synthesis and structure elucidation of hydrazones derived from *N*-(2,4-dimethylphenyl)-3-oxobutanamide

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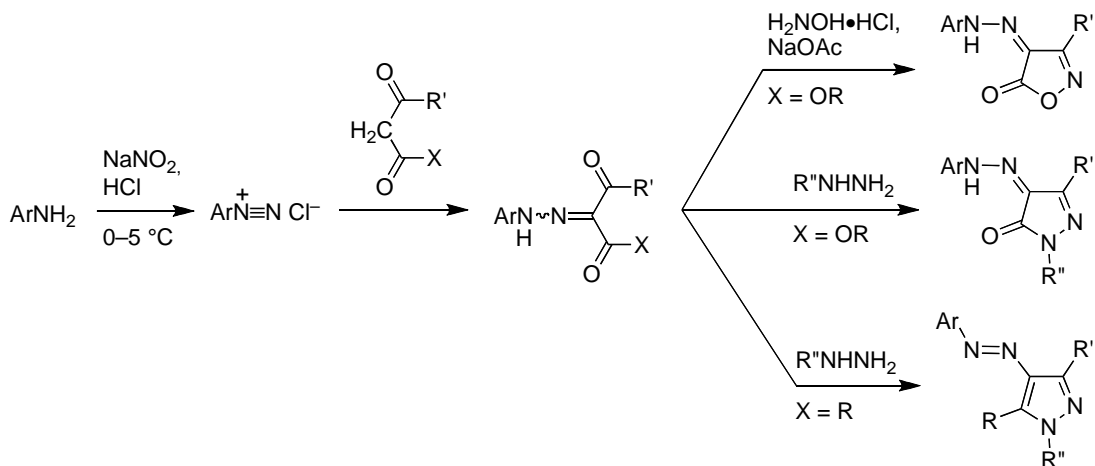
### Abstract

Diazonium salts derived from amines **1** (sulfanilic acid, 4-nitroaniline, 4-aminosalicylic acid, sulfanilamide, sulfamethoxazole, sulfathiazole, sulfapyridine, sulfamerazine) were coupled with *N*-(2,4-dimethylphenyl)-3-oxobutanamide (**2**) resulting in the formation of hydrazones **3a–h**.

**Keywords:** Azo coupling, hydrazones

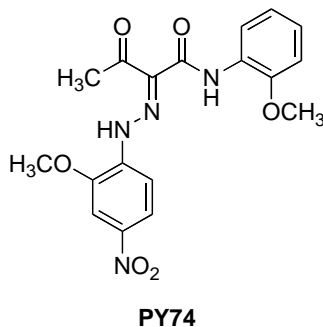
### Introduction

Coupling products of diazonium salts with aliphatic active hydrogen compounds are widely used as intermediates for the synthesis of a large number of heterocyclic compounds. Pyrazoles,<sup>1,2</sup> isoxazolone,<sup>3</sup> 2-pyrazoline-5-one<sup>4,5</sup> can be obtained by cyclization of coupling products with substituted hydrazine or hydroxylamine, respectively (Scheme 1). Both hydrazones<sup>5,6-9</sup> and their cyclization compounds<sup>2-4</sup> possess important biological and pharmacological properties.

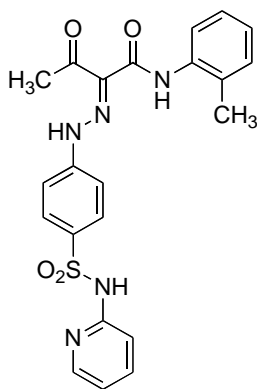


**Scheme 1.** General synthetic routes for the formation of azo compounds and hydrazones.

Azo dyes are among the most important synthetic coloring agents but are regarded as potential carcinogens<sup>10</sup> due to their metabolism; the reduction of the azo group affects their toxicity, mutagenicity, and carcinogenicity.<sup>10-13</sup> Hydrazone dyes are considered non-genotoxic and non-mutagenic; e.g., 2-[(2-methoxy-5-nitrophenyl)hydrazono]-*N*-(2-methoxyphenyl)-3-oxobutanamide (PY74) is a hydrazone pigment used in yellow tattoo inks. The metabolism of PY74<sup>14</sup> and compounds containing azo group<sup>15,16</sup> has been investigated using rat liver and human liver microsomes.



The hydrazone product obtained by azo coupling of the diazonium salt of sulfapyridine with *N*-(2-methylphenyl)-3-oxobutanamide has been found to be an HIV integrase inhibitor.<sup>17</sup>

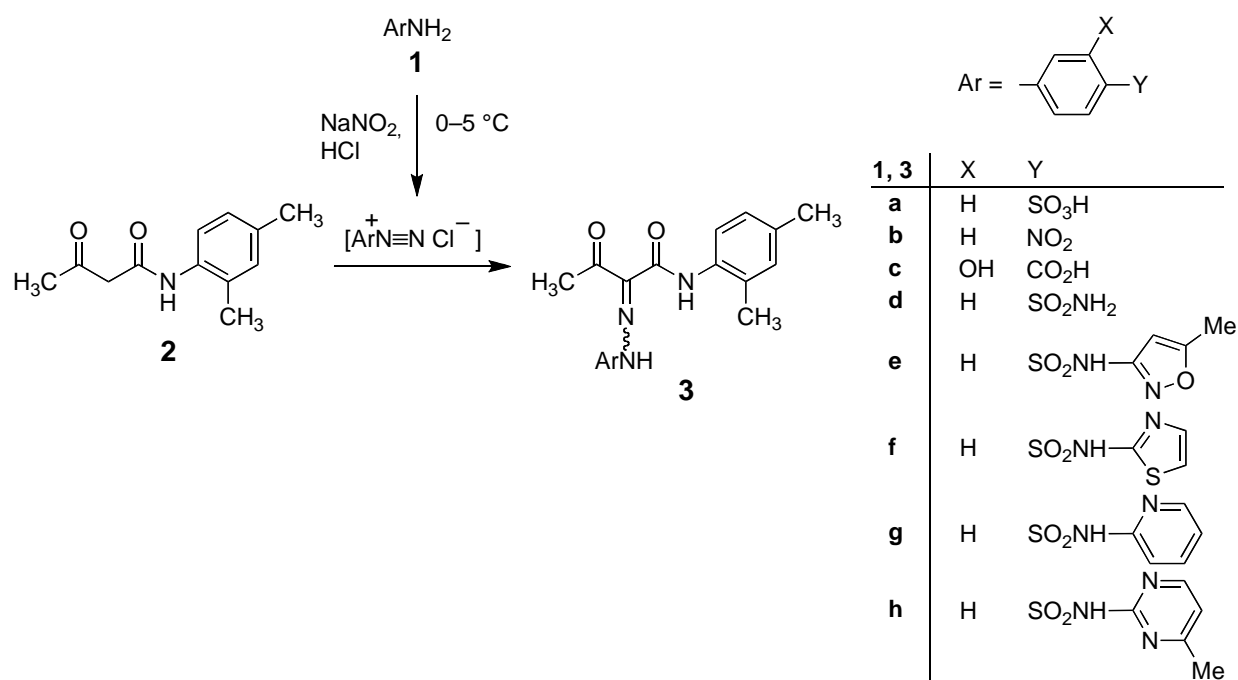


Earlier reports indicate that diazonium salts of certain aromatic amines such as sulfaguanidine<sup>18</sup> and sulfanilamide<sup>19</sup> have been coupled with compounds possessing active hydrogen. Furthermore, sulfanilamide derivatives have been reported to possess antibacterial activity.<sup>20</sup> The present study reports on the synthesis of new coupling products **3a-h**, which were obtained from diazonium salts derived from amines **1** (sulfanilic acid, 4-nitroaniline, 4-aminosalicylic acid, sulfanilamide, sulfamethoxazole, sulfathiazole, sulfapyridine, sulfamerazine) with *N*-(2,4-dimethylphenyl)-3-oxobutanamide (**2**).

## Results and Discussion

The diazonium salts derived from anilines **1** (sulfanilic acid, 4-nitroaniline, 4-aminosalicylic acid, sulfanilamide, sulfamethoxazole, sulfathiazole, sulfapyridine, sulfamerazine) were coupled with *N*-(2,4-dimethylphenyl)-3-oxobutanamide (**2**) in aqueous ethanol containing sodium acetate resulting in the formation of hydrazones **3a–h** (Scheme 2).

The UV spectra of products **3** show three ranges of absorption maxima at 203–207, 235–271, and 376–393 nm, except compounds **3e** and **3f** which had four absorption maxima. Absorption bands attributed to an azo function between 332–360 nm<sup>21–22</sup> and above 400 nm<sup>23</sup> are missing. Thus, the observation of bands at 376–393 nm is indicative of the hydrazone form of compounds **3a–h**.<sup>6,9,24–26</sup>



**Scheme 2.** Preparation of [2-[1-(2,4-dimethylphenylamino)-1,3-dioxobutan-2-ylidene]hydrazinyl]benzenes (**3a–h**).

The amide proton (-NH-C=O) exhibits a singlet at  $\delta$  10.98–11.16, the hydrazone proton (-CH=N-NH-) shows a singlet at  $\delta$  13.90–14.32; both signal ranges are in agreement with the literature.<sup>18,27–29</sup> Furthermore, in the <sup>1</sup>H-NMR spectra of compounds **3a–h**, signals arising from a >CH-N=N- moiety are expected at 3.00–4.00 ppm,<sup>23,25</sup> but were not observed. These findings support the hydrazone structure of the products.

The APCI-MS spectra of **3a–h** show molecular ion peaks (M<sup>+</sup>) confirming their molecular weight; common characteristic fragment ions result from cleavage of the amide bond resulting in

2,4-dimethylanilinium ion ( $m/z$  122) and the complementing 2-[(2-arylhydrazono)-3-oxo-butylidene]oxonium ion ( $m/z$   $M+1-122$ ).

## Experimental Section

**General Procedures.** All chemicals and solvents were commercially acquired. Melting points were determined with a Schmelzpunktbestimmer SMP II. The UV spectra were measured with a Shimadzu UV-2100 S. The IR spectra were obtained with a Shimadzu FTIR-8400.  $^1\text{H-NMR}$  spectra in  $\text{DMSO-}d_6$  were recorded on a Bruker Avance-DPX-400 spectrometer (400 MHz). APCI-MS was performed using an Agilent 1100 MSD spectrometer at 100 eV (positive polarity). All new compounds were analyzed for C, H, N (Leco CHNS 932).

$^1\text{H NMR}$ , APCI-MS, and elemental analyses were provided by the Scientific and Technical Research Council of Turkey, (TÜBİTAK).

### [2-[1-(2,4-Dimethylphenylamino)-1,3-dioxobutan-2-ylidene]hydrazinyl]benzenes (3a-h).

#### General procedure

To the cooled (0–5 °C) solution of amine **1** (0.01 mol) in ethanol (50 mL) and hydrochloric acid (4%; 40 mL) was added an ice-cold solution of sodium nitrite (7%; 10 mL). The reaction mixture was then poured into a solution of *N*-(2,4-dimethylphenyl)-3-oxo-butanamide (**2**; 2.05 g, 0.01 mol) and sodium acetate (60 g, 0.73 mol) in ethanol (50%; 50 mL) under vigorous stirring. The precipitated solid was collected, washed with water, air-dried at room temperature, and washed with ethanol to give **3**.

$^1\text{H NMR}$  spectra: H' refers to the X,Y-substituted benzene ring Ar (cf. Scheme 2).

**4-[2-[1-(2,4-Dimethylphenylamino)-1,3-dioxobutan-2-ylidene]hydrazinyl]benzenesulfonic acid (3a).** Yellow needles (3.46 g, 89%); mp 357 °C (EtOH; decomp.). UV  $\lambda_{\text{max}}$  (EtOH): nm (log  $\epsilon$ ) 376 (4.53), 250 (4.26), 204 (4.45). IR (KBr):  $\nu_{\text{max}}$  ( $\text{cm}^{-1}$ ) 3592, 3511 (OH), 3170–3130 (NH), 1680 (ketone C=O), 1600 (amide C=O), 1315–1135 (S=O).  $^1\text{H NMR}$  ( $\text{DMSO-}d_6$ ):  $\delta$  2.26 (3H, s, CH<sub>3</sub>), 2.29 (3H, s, CH<sub>3</sub>), 2.55 (3H, s, COCH<sub>3</sub>), 7.04 (1H, d,  $^3J_{6,5} = 8.2$  Hz, H6), 7.11 (1H, s, H3), 7.45–7.70 (4H, m, H2', H3', H5', H6'), 7.96 (1H, d,  $^3J_{5,6} = 8.2$  Hz, H5), 11.16 (1H, s, CONH), 14.32 (1H, s, NNH). APCI-MS:  $m/z$  (%) 390 (100)  $[\text{M}+1]^+$ , 371 (3.7), 269 (24), 152 (4.4), 122 (37). Anal. calcd. for  $\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_5\text{S}\cdot 2\text{H}_2\text{O}$ : C, 50.81; H, 5.45; N, 9.88; S, 7.54. Found: C, 50.25; H, 5.24; N, 10.03; S, 7.23.

***N*-(2,4-Dimethylphenyl)-2-[2-(4-nitrophenyl)hydrazono]-3-oxobutanamide (3b).** Yellow needles (2.76 g, 78%); mp 222 °C (EtOH). UV  $\lambda_{\text{max}}$  (EtOH): nm (log  $\epsilon$ ) 392 (4.40), 235 (4.06), 203 (4.34). IR (KBr):  $\nu_{\text{max}}$  ( $\text{cm}^{-1}$ ): 3220, 3130 (NH), 1663 (ketone C=O), 1595 (amide C=O).  $^1\text{H NMR}$  ( $\text{DMSO-}d_6$ ):  $\delta$  2.27 (3H, s, CH<sub>3</sub>), 2.29 (3H, s, CH<sub>3</sub>), 2.57 (3H, s, COCH<sub>3</sub>), 7.05 (1H, d,  $^3J_{6,5} = 8.8$  Hz, H6), 7.11 (1H, s, H3), 7.71–8.30 (5H, m, H5, H2', H3', H5', H6'), 11.00 (1H, s, CONH), 13.90 (1H, s, NNH). APCI-MS:  $m/z$  (%) 355 (100)  $[\text{M}+1]^+$  234 (31.5), 139 (5.0), 122

(39.1). Anal. calcd. for  $C_{18}H_{18}N_4O_4$ : C, 61.01; H, 5.12; N, 15.81. Found: C, 60.71; H, 4.98; N, 16.21.

**4-[2-[1-(2,4-Dimethylphenylamino)-1,3-dioxobutan-2-ylidene]hydrazinyl]-2-hydroxybenzoic acid (3c).** Yellow needles (2.92 g, 79%); mp 245 °C (EtOH). UV  $\lambda_{max}$ . (EtOH): nm (log  $\epsilon$ ) 386 (4.55), 271 (4.24), 206 (4.60). IR (KBr):  $\nu_{max}$ . ( $cm^{-1}$ ): 3220, 3130 (NH), 1680 (carboxylic acid C=O), 1660 (ketone C=O), 1638 (amide C=O).  $^1H$  NMR (DMSO- $d_6$ ):  $\delta$  2.26 (3H, s, CH<sub>3</sub>), 2.28 (3H, s, CH<sub>3</sub>), 2.54 (3H, s, COCH<sub>3</sub>), 7.00 (1H, s, H2'), 7.01 (1H, d,  $^3J_{6,5} = 8.0$  Hz, H6), 7.05 (1H, d,  $^3J_{6',5'} = 10.0$  Hz, H6'), 7.09 (1H, s, H3), 7.79 (1H, d,  $^3J_{5',6'} = 9.2$  Hz, H5'), 7.93 (1H, d,  $^3J_{5,6} = 8.2$  Hz, H5), 11.04 (1H, s, CONH), 13.90 (1H, s, NNH). APCI-MS:  $m/z$  (%): 370 (100) [M+1]<sup>+</sup>, 355 (16.6), 249 (53.9), 122 (23.6). Anal. calcd. for  $C_{19}H_{19}N_3O_5 \cdot H_2O$ : C, 58.91; H, 5.46; N, 10.85. Found: C, 58.73; H, 4.73; N, 11.12.

**N-(2,4-Dimethylphenyl)-3-oxo-2-[2-(4-sulfamoylphenyl)hydrazono]butanamide (3d).**

Yellow needles (3.06 g, 79%); mp 260 °C (EtOH). UV  $\lambda_{max}$ . (EtOH): nm (log  $\epsilon$ ) 376 (4.17), 236 (3.92), 203 (4.33). IR (KBr):  $\nu_{max}$ . ( $cm^{-1}$ ): 3317, 3234, 3170 (NH), 1663 (ketone C=O), 1596 (amide C=O), 1151 (S=O).  $^1H$  NMR (DMSO  $d_6$ ):  $\delta$  2.27 (3H, s, CH<sub>3</sub>), 2.29 (3H, s, CH<sub>3</sub>), 2.57 (3H, s, COCH<sub>3</sub>), 7.05 (1H, d,  $^3J_{6,5} = 8.2$  Hz, H6), 7.10 (1H, s, H3), 7.34 (1H, s, SO<sub>2</sub>NH), 7.66–7.90 (4H, m, H2', H3', H5', H6'), 7.94 (1H, d,  $^3J_{5,6} = 8.2$  Hz, H5), 11.05 (1H, s, CONH), 14.10 (1H, s, NNH). APCI-MS:  $m/z$  (%): 389 (28.2) [M+1]<sup>+</sup>, 372 (7.3), 283 (43.9), 268 (91.6), 122 (100). Anal. calcd. for  $C_{18}H_{20}N_4O_4S$ : C, 55.66; H, 5.19; N, 14.42; S, 8.25. Found: C, 55.83; H, 5.26; N, 14.19; S, 7.84.

**N-(2,4-Dimethylphenyl)-2-[2-[4-[N-(5-methylisoxazol-3-yl)sulfamoyl]phenyl]hydrazono]-3-oxobutanamide (3e).** Yellow needles (4.03 g, 86%); mp 215 °C (EtOH). UV  $\lambda_{max}$ . (EtOH): nm (log  $\epsilon$ ) 376 (4.44), 249 (4.15), 237 (4.16), 204 (8.98). IR (KBr):  $\nu_{max}$  ( $cm^{-1}$ ): 3220, 3130 (NH), 1667 (ketone C=O), 1614 (amide C=O), 1137 (S=O).  $^1H$  NMR (DMSO- $d_6$ ):  $\delta$  2.26 (3H, s, CH<sub>3</sub>), 2.28 (3H, s, CH<sub>3</sub>), 2.30 (3H, s, CH<sub>3</sub> at isoxazole), 2.55 (3H, s, COCH<sub>3</sub>), 6.15 (1H, s, CH isoxazole), 7.04 (1H, d,  $^3J_{6,5} = 8.3$  Hz, H6), 7.10 (1H, s, H3), 7.67–7.92 (4H, m, H2', H3', H5', H6'), 7.92 (1H, d,  $^3J_{5,6} = 8.2$  Hz, H5), 10.98 (1H, s, CONH), 11.40 (1H, s, SO<sub>2</sub>NH), 14.02 (1H, s, NNH). APCI-MS:  $m/z$  (%) 470 (100) [M+1]<sup>+</sup>, 372 (19.6), 349 (48.7), 254 (26.5), 122 (22.0). Anal. calcd. for  $C_{22}H_{23}N_5O_5S$ : C, 56.28; H, 4.94; N, 14.92; S, 6.83. Found: C, 56.19; H, 4.52; N, 14.98; S, 6.63.

**N-(2,4-Dimethylphenyl)-3-oxo-2-[2-[4-(N-thiazol-2-ylsulfamoyl)phenyl]hydrazono]butanamide (3f).** Yellow needles (3.34 g, 71%); mp 280 °C (EtOH). UV  $\lambda_{max}$ . (EtOH): nm (log  $\epsilon$ ) 381 (4.31), 271 (4.08), 251 (4.03), 202 (4.39). IR (KBr):  $\nu_{max}$ . ( $cm^{-1}$ ): 3220, 3130 (NH), 1665 (ketone C=O), 1630 (amide C=O), 1149 (S=O).  $^1H$ -NMR (DMSO- $d_6$ ):  $\delta$  2.26 (3H, s, CH<sub>3</sub>), 2.28 (3H, s, CH<sub>3</sub>), 2.55 (3H, s, COCH<sub>3</sub>), 6.84 (1H, d,  $^3J_{5,4} = 4.5$  Hz, H5 thiazole), 7.03 (1H, d,  $^3J_{6,5} = 8.3$  Hz, H6), 7.09 (1H, s, H3), 7.26 (1H, d,  $^3J_{4,5} = 4.6$  Hz, H4 thiazole), 7.62–7.88 (4H, m H2', H3', H5', H6'), 7.93 (1H, d,  $^3J_{5,6} = 8.2$  Hz, H5), 11.03 (1H, s, CONH), 12.75 (1H, s, SO<sub>2</sub>NH), 14.11 (1H, s, NNH). APCI-MS:  $m/z$  (%) 472 (100) [M+1]<sup>+</sup>, 372 (9.5), 351 (17.0), 256 (10.9), 122 (7.3). Anal. calcd. for  $C_{21}H_{21}N_5O_4S_2 \cdot 1/2H_2O$ : C, 52.50; H, 4.58; N, 14.58; S, 13.33. Found: C, 52.13; H, 5.14; N, 14.83; S, 13.38.

***N*-(2,4-Dimethylphenyl)-3-oxo-2-[2-[4-(*N*-pyridin-2-ylsulfamoyl)phenyl]hydrazono]butanamide (3g).** Yellow needles (3.12 g, 67%); mp 240 °C (EtOH). UV  $\lambda_{\max}$ . (EtOH): nm (log  $\epsilon$ ): 375 (4.11), 247 (3.93), 203 (4.31). IR (KBr):  $\nu_{\max}$ . ( $\text{cm}^{-1}$ ): 3220, 3130 (NH), 1668 (ketone C=O), 1633 (amide C=O), 1139 (S=O).  $^1\text{H}$  NMR (DMSO- $d_6$ ):  $\delta$  2.26 (3H, s, CH<sub>3</sub>), 2.28 (3H, s, CH<sub>3</sub>), 2.54 (3H, s, COCH<sub>3</sub>), 6.88 (1H, t, H4 pyridine), 7.03 (1H, d,  $^3J_{6,5}$  = 8.7 Hz, H6), 7.09 (1H, s, H3), 7.16 (1H, d,  $^3J_{5,6}$  = 8.3 Hz, H5 pyridine), 7.62-7.93 (5H, m, H3 pyridine, H2', H3', H5', H6'), 7.97 (1H, d,  $^3J_{5,6}$  = 8.3 Hz, H5), 8.01 (1H, d,  $^3J_{6,5}$  = 4.4 Hz, H6 pyridine), 11.02 (1H, s, CONH), 11.90 (1H, s, SO<sub>2</sub>NH), 14.08 (1H, s, NNH). APCI-MS:  $m/z$  466 (100) [M+1]<sup>+</sup>, 345 (3.6), 318 (1.4), 268 (2.2), 250 (4.2), 122 (4.1). Anal. calcd. for C<sub>23</sub>H<sub>23</sub>N<sub>5</sub>O<sub>4</sub>S·H<sub>2</sub>O: C, 57.13; H, 5.21; N, 14.48; S, 6.63. Found: C, 57.95; H, 4.81; N, 14.81; S, 6.85.

***N*-(2,4-Dimethylphenyl)-2-[2-[4-[*N*-(4-methylpyrimidin-2-yl)sulfamoyl]phenyl]hydrazono]-3-oxobutanamide (3h).** Yellow needles (3.02 g, 63%); mp 288 °C. UV  $\lambda_{\max}$ . (EtOH): nm (log  $\epsilon$ ): 376 (4.49), 253 (4.28), 203 (4.53). IR (KBr):  $\nu_{\max}$ . ( $\text{cm}^{-1}$ ): 3250, 3120 (NH), 1668 (ketone C=O), 1615 (amide C=O), 1343, 1175 (S=O).  $^1\text{H}$  NMR (DMSO- $d_6$ ):  $\delta$  2.26 (3H, s, CH<sub>3</sub>), 2.28 (3H, s, CH<sub>3</sub>), 2.33 (3H, s, CH<sub>3</sub> at pyrimidine), 2.55 (3H, s, COCH<sub>3</sub>), 6.90 (1H, d,  $^3J_{6,5}$  = 5.0 Hz, H6 pyrimidine), 7.00 (1H, d,  $^3J_{6,5}$  = 8.2 Hz, H6), 7.09 (1H, s, H3), 7.65–8.06 (5H, m H5, H2', H3', H5', H6'), 8.32 (1H, d,  $^3J_{5,6}$  = 5.0 Hz, H5 pyrimidine), 11.01 (1H, s, CONH), 11.67 (1H, s, SO<sub>2</sub>NH), 14.07 (1H, s, NNH). APCI-MS:  $m/z$  (%) 481 (100) [M+1]<sup>+</sup>, 467 (4.0), 332 (6.3), 265 (4.1). Anal. calcd. for C<sub>23</sub>H<sub>24</sub>N<sub>6</sub>O<sub>4</sub>S: C, 57.49; H, 5.03; N, 17.49; S, 6.67. Found: C, 57.36; H, 5.73; N, 17.67; S, 6.59.

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