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Short Note

N,*N*'-Bis-(2,5-dimethyl-3-oxo-1-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)phthalamide

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Abstract: The title compound, *N*,*N*'-bis-(2,5-dimethyl-3-oxo-1-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)phthalamide has been synthesized by reactions of *o*-phthaloyl chloride with 4-amino-2,5-dimethyl-1-phenyl-3-oxo-1*H*-pyrazolone in acetonitrile. The structure of the new compound was characterized by FT-IR, ^{1}H NMR, ^{13}C NMR and mass spectroscopic techniques. Physical parameters of the compound such as melting point, solubility, λ_{max} were examined.

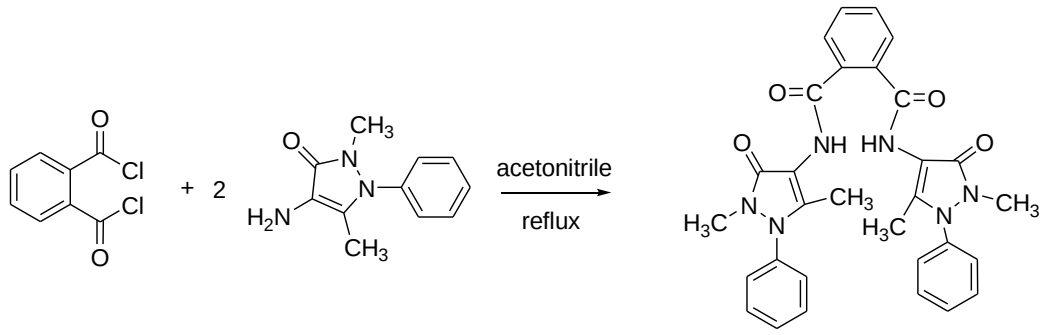
Keywords: *N*-substituted amide; 4-amino-2,5-dimethyl-1-phenyl-3-oxo-1*H*-pyrazolone; *o*-phthaloyl chloride

Introduction

The pyrazole ring has been known as an important framework in a large number of compounds possessing pharmaceutical and agrochemical properties [1–3]. In addition, some substituted pyrazolines have constituted an important class of conjugated nitrogen-containing fluorescent compounds [4]. Functionalized *N*-arylpyrazoles have been shown to exhibit antihyperglycemic, analgesic, anti-inflammatory, sedative, and hypnotic activities [5]. Some phthalic acid diamide derivatives have been used as agricultural and horticultural insecticides [6].

In view of this, we report the convenient preparation of a new representative of this type of compounds, combining both of the above-mentioned structural elements.

Scheme 1. Synthesis of *N*,*N*'-bis-(2,5-dimethyl-3-oxo-1-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)phthalamide.



Experimental

Reagents and Techniques

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE DPX NMR spectrometer operating at 400 and 101.6 MHz. Infrared absorption spectra were obtained from a Perkin Elmer BX-II Spectrum 100 and are reported in cm^{-1} units. Melting points were measured on an Electro Thermal IA 9100 apparatus using a capillary tube. LC mass spectra were obtained on an AGILENT 1100 MSD spectrometer with an ion source temperature of 240 ℃. *o*-Phthaloyl chloride, 4-amino-2,5-dimethyl-1-phenyl-3-oxo-1*H*-pyrazolone, acetonitrile, tetrahydrofuran and acetone were purchased from Merck.

Preparation of the Title Compound

A solution of *o*-phthaloyl chloride (1.01 g, 5 mmol) in acetonitrile (20 mL) was added dropwise to a solution of 4-amino-2,5-dimethyl-1-phenyl-3-oxo-1*H*-pyrazolone (2.03 g, 10 mmol) in acetonitrile (20 mL). The mixture was refluxed with stirring for 3 h. The progress of the reaction was controlled by TLC. After cooling the reaction mixture to ambient temperature, the solid was filtered off and recrystallized from tetrahydrofuran/acetone to give the product. The physical and spectral data of the compound are as follows: colorless crystal, yield 2.68 g (88%), melting point: 204–206 ℃.

UV-Vis, λ_{max}: 281 nm (in Me_2SO);

FT-IR : ν_{max} (cm^{-1}): 3496 (N-H stretching), 3029 (aromatic C-H stretching), 2986 (C-H aliphatic), 1675 (amide, C=O stretching), 1783, 1713 (C=O, pyrazolone), 1490 (N-CO);

^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 6.43 (s, 2H, N-H), 8.01 (t, J = 4.3 Hz, 2H, $C_{phtaloyl}$-H), 7.95 (d, J = 3.8 Hz, 2H, $C_{phtaloyl}$-H);), 7.92 (t, J = 5.1 Hz, 4H, $C_{phen.}$-H), 7.54 (t, J = 3.5 Hz, 2H, $C_{phen.}$-H), 7.39 (d, J = 3.2, 4H, $C_{phen.}$-H), 3.43 (s, 6H, N-CH_3), 2.08 (s, 6H, C-CH_3);

^{13}C-NMR (100 MHz, DMSO-d_6) δ ppm: 168.29 (C=O), 161.49 (C=O), 135.33, 132.81, 130.59 ($C_{phtaloyl}$), 136.21, 124.83, 128.69, 126.12 (C_{phenyl}), 154.90, 100.50 ($C_{pyrazolone}$), 35.83 (N-CH_3), 10.87 (C-CH_3);

Anal. Calcd. for $C_{30}H_{28}N_6O_4$: C, 67.15%; H, 5.26%; N, 15.66%. Found: C, 67.06%; H, 5.31%; N, 15.67%.

Acknowledgements

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References

1. Ali, M.A.; Siddiqui, M.S.A.A. Synthesis, structural activity relationship and anti-tubercular activity of novel pyrazoline derivatives. *Eur. J. Med. Chem*. **2007**, *42*, 268–275.
2. Azarifar, D.; Shaebanzadeh, M. Synthesis and characterization of new 3,5-dinaphthyl substituted 2-pyrazolines and study of their antimicrobial activities. *Molecules* **2002**, *7*, 885–895.
3. Abunada, N.M.; Hassaneen, H.M.; Kandile, N.G.; Miqdad, O.M. Synthesis and biological activity of some new pyrazoline and pyrolo[3,4-c]pyrazole-4,6-dione derivatives: Reaction of nitrilimines with some dipolarophiles. *Molecules* **2008**, *13*, 1011–1024.
4. Hasan, A.; Abbas, A.; Akhtar, M.N. Synthesis, characterization and fluoresent property evaluation of 1,3,5-triaryl-2-pyrazolines. *Molecules* **2011**, *16*, 7789–7802.
5. Amir, M.; Kumar, H.; Khan, S.A. Synthesis and pharmacological evaluation of pyrazoline derivatives as new anti-inflammatory and analgesic agents. *Bioorg. Med. Chem. Lett.* **2008**, *18*, 918–922.
6. Feng, M.L.; Li, Y.-F.; Zhu, H.-J.; Zhao, L.; Xi, B.-B.; Ni, J.-P. Synthesis, insecticidal activity and structure activity relation of trifluoromethyl containing phthalic acid diamide structure. *J. Agr. Food Chem.* **2010**, *58*, 10999–11006.