Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supplementary Information

Imidazo[1,5-*a*]pyridine-1-ylalkylalcohols: synthesis via intramolecular cyclization of *N*-thioacyl 1,2-aminoalcohols and their silyl ethers and molecular structures†

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X-ray Structure Analyses. The measurement of **8a**, **2e** and **9g** was carried out on a Rigaku/MSC Mercury CCD diffractometer with graphite-monochlomates Mo-Ka radiation (l = 0.71069 Å). Reflection data were collected at 123-203 K using a Rigaku XR-TCS-2-050 temperature controller. The structure was solved by direct methods (SIR97)[S1)] and refined by full-matrix least-squares procedures (SHELXL-97)[S2)] using the Yadokari-XG 2009[S3)]. The X-ray quality crystal was obtained by slow diffusion of hexane into CH_2Cl_2 solution of **8a**, **2e** and **9g**. The crystal was cut from the grown crystals and was attached to the tip of a MiTeGen MicroMount™. Crystal data and measurement description are summarized in Tables S1–S3.

References

S1) A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori and R. Spagna, *J. Appl. Cryst.*, 1999, **32**, 115.

S2) G. M. Sheldrick, SHELXL-97, *A Program for the Refinement of Crystal Structures*; University of Göttingen: Göttingen, Germany, 1997.

S3) (*a*) K. Wakita, Yadokari-XG, *Software for Crystal Structure Analyses*, 2001; (*b*) C. Kabuto, S. Akine, T. Nemoto, E. Kwon, *J. Cryst. Soc. Jpn.*, 2009, **51**, 218.

Table S1. Crystal data and structure refinement for **8a**.

Identification code	en013-3	
Empirical formula	$C_{41}H_{32}N_4$	
Formula weight	580.71	
Temperature	153(2) K	
Wavelength	0.71070 Å	
Crystal system	Monoclinic	
Space group	*C*2/*c*	
Unit cell dimensions	a = 22.232(18) Å	α = 90°.
	b = 10.636(7) Å	β = 133.986(7)°.
	c = 17.824(14) Å	γ = 90°.
Volume	3032(4) Å^3	
Z	4	
Density (calculated)	1.272 Mg/m^3	
Absorption coefficient	0.075 mm^{-1}	
F(000)	1224	
Crystal size	0.29 x 0.17 x 0.03 mm^3	
Theta range for data collection	3.12 to 27.48°.	
Index ranges	-28<=*h*<=22, -13<=*k*<=11, -23<=*l*<=22	
Reflections collected	12275	
Independent reflections	3463 [R(int) = 0.0766]	
Completeness to θ = 27.48°	99.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.998 and 0.988	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3463 / 0 / 205	
Final R indices [I>2σ (I)][a]	*R1* = 0.1049, *wR2* = 0.1825	
R indices (all data) [b]	*R1* = 0.1551, *wR2* = 0.2078	
Goodness-of-fit on F^2 [c]	1.000	
Largest diff. peak and hole	0.305 and -0.182 e.Å^{-3}	

[a] $R1 = \Sigma ||F_0| - |F_c|| / \Sigma |F_0|$. [b] $wR2 = [\Sigma \{w(F_0^2 - F_e^2)^2\} / \Sigma w(F_0^2)^2]^{1/2}$, $w = 1/[\sigma^2 F_0^2 + (aP)^2 + bP]$ (a and b are constants suggested by the refinement program; $P = [\max(F_0^2, 0) + 2F_e^2]/3$). [c] GOF = $[\Sigma w(F_0^2 - F_e^2)^2/(N_{obs} - N_{params})]^{1/2}$.

Table S2. Crystal data and structure refinement for **2e**.

Identification code	en091	
Empirical formula	$C_{18}H_{14}N_4O$	
Formula weight	302.33	
Temperature	203(2) K	
Wavelength	0.71070 Å	
Crystal system	Orthorhombic	
Space group	*Pna2₁*	
Unit cell dimensions	a = 25.97(2) Å	α = 90°.
	b = 4.887(4) Å	β = 90°.
	c = 11.168(9) Å	γ = 90°.
Volume	1417.7(19) $Å^3$	
Z	4	
Density (calculated)	1.417 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	632	
Crystal size	0.29 x 0.26 x 0.14 mm^3	
Theta range for data collection	4.24 to 27.50°.	
Index ranges	-33<=*h*<=33, -6<=*k*<=4, -10<=*l*<=14	
Reflections collected	10824	
Independent reflections	1684 [R(int) = 0.0755]	
Completeness to z = 27.50°	98.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.989 and 0.975	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1684 / 1 / 209	
Final R indices [I>2σ(I)][a]	*R1* = 0.0522, *wR2* = 0.1140	
R indices (all data) [b]	*R1* = 0.0532, *wR2* = 0.1145	
Absolute structure parameter	0(10)	
Goodness-of-fit on F^2 [c]	1.000	
Largest diff. peak and hole	0.372 and -0.214 e.$Å^{-3}$	

[a] $R1 = \Sigma ||F_0| - |F_c|| / \Sigma |F_0|$. [b] $wR2 = [\Sigma \{w(F_0^2 - F_e^2)^2\} / \Sigma w(F_0^2)^2]^{1/2}$, $w = 1/[\sigma^2 F_0^2 + (aP)^2 + bP]$ (a and b are constants suggested by the refinement program; $P = [\max(F_0^2,0) + 2F_e^2]/3$). [c] $GOF = [\Sigma w(F_0^2 - F_e^2)^2/(N_{obs} - N_{params})]^{1/2}$.

Table S3. Crystal data and structure refinement for **9g•CH_2Cl_2**.

Identification code	en064-2	
Empirical formula	$C_{28}H_{22}Cl_2N_2O$	
Formula weight	473.38	
Temperature	203(2) K	
Wavelength	0.71070 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 8.513(2) Å	α = 90°.
	b = 23.170(6) Å	β = 103.189(3)°.
	c = 11.837(3) Å	γ = 90°.
Volume	2273.1(11) $Å^3$	
Z	4	
Density (calculated)	1.383 Mg/m^3	
Absorption coefficient	0.310 mm^{-1}	
F(000)	984	
Crystal size	0.34 x 0.34 x 0.23 mm^3	
Theta range for data collection	3.02 to 27.50°.	
Index ranges	-11<=*h*<=11, -29<=*k*<=30, -15<=*l*<=13	
Reflections collected	18043	
Independent reflections	5151 [R(int) = 0.0297]	
Completeness to z = 27.50°	98.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.986 and 0.970	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5151 / 0 / 301	
Final R indices [I>2σ(I)][a]	*R1* = 0.0587, *wR2* = 0.1159	
R indices (all data)[b]	*R1* = 0.0637, *wR2* = 0.1189	
Goodness-of-fit on F^2 [c]	1.000	
Largest diff. peak and hole	0.605 and -0.662 e.$Å^{-3}$	

[a] $R1 = \Sigma ||F_0| - |F_c|| / \Sigma |F_0|$. [b] $wR2 = [\Sigma \{w(F_0^2 - F_e^2)^2\} / \Sigma w(F_0^2)^2]^{1/2}$, $w = 1/[\sigma^2 F_0^2 + (aP)^2 + bP]$ (a and b are constants suggested by the refinement program; $P = [\max(F_0^2,0) + 2F_e^2]/3$). [c] $GOF = [\Sigma w(F_0^2 - F_e^2)^2/(N_{obs} - N_{params})]^{1/2}$.