Supporting material for:

SUPPORTING INFORMATION

Cyclization of 1-(2-Alkynylphenyl)-3,3-dialkyltriazenes: A Convenient, High-Yield Synthesis of Substituted Cinnolines and Isoindazoles

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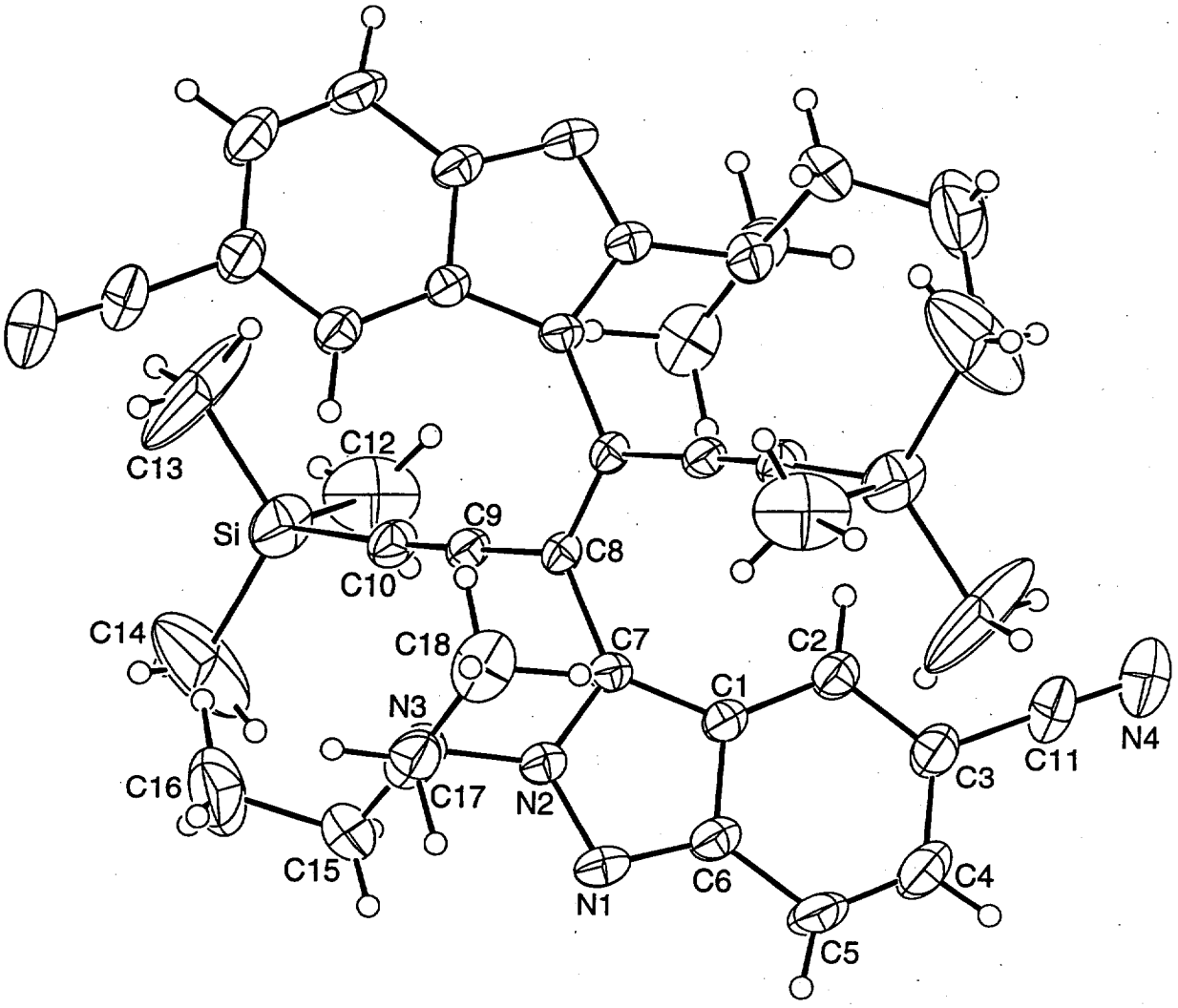
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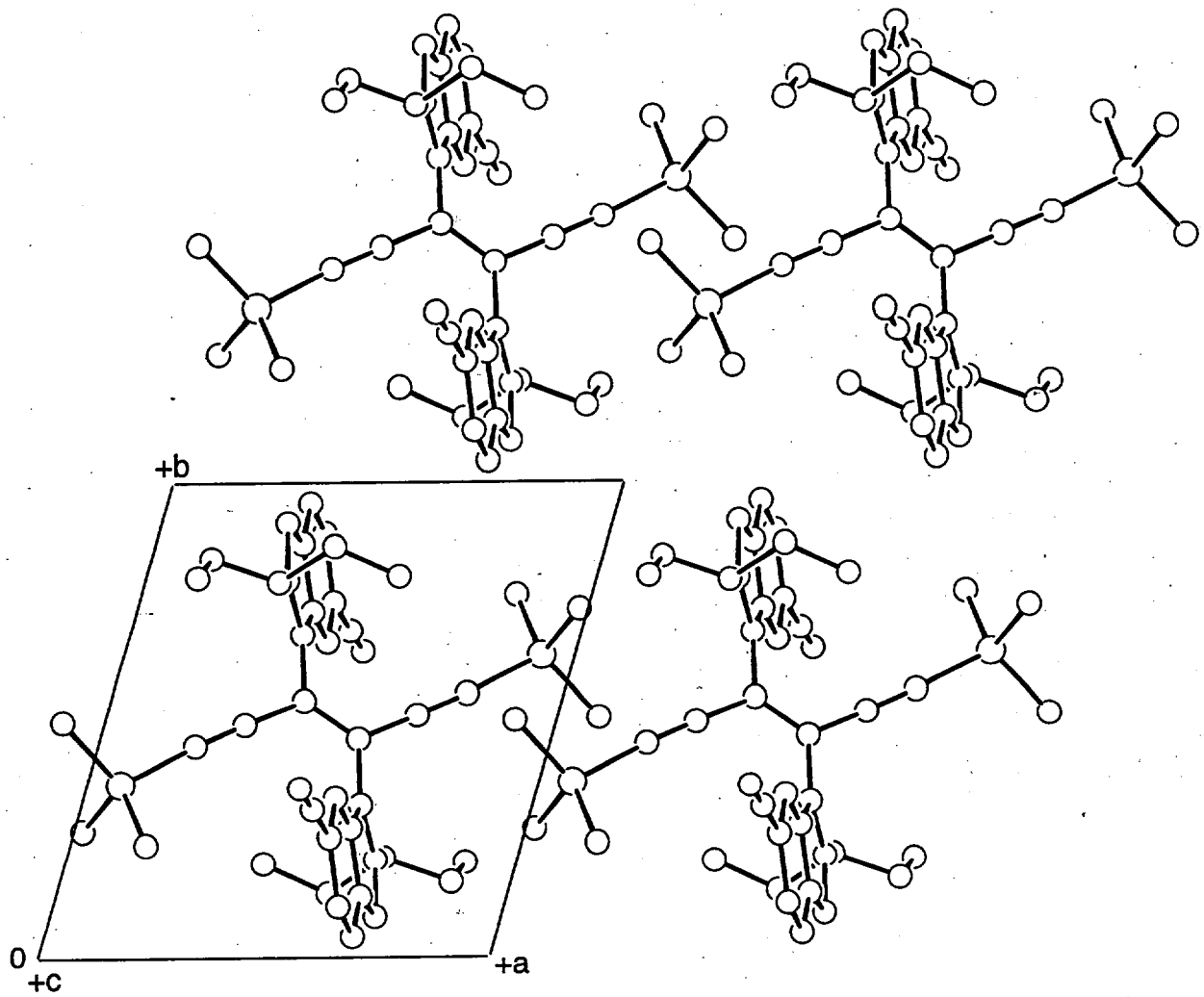
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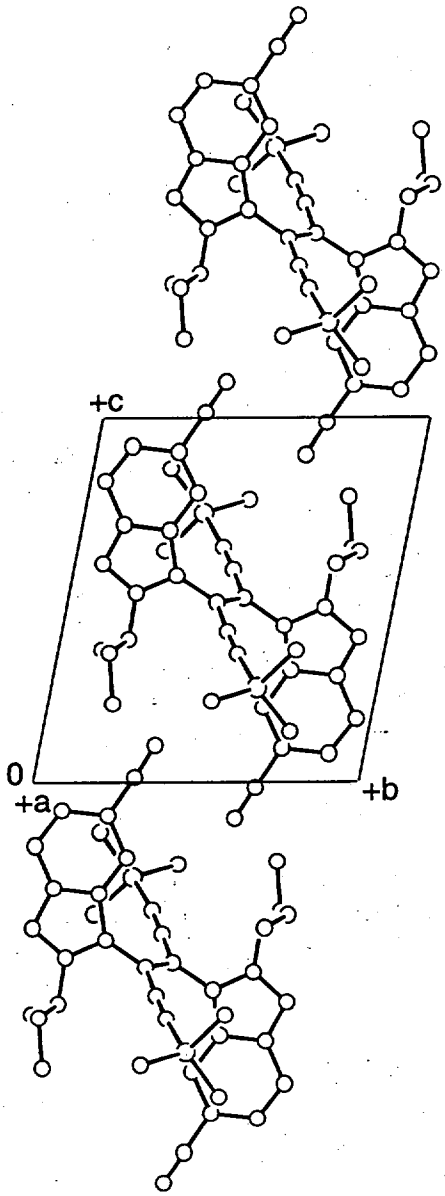
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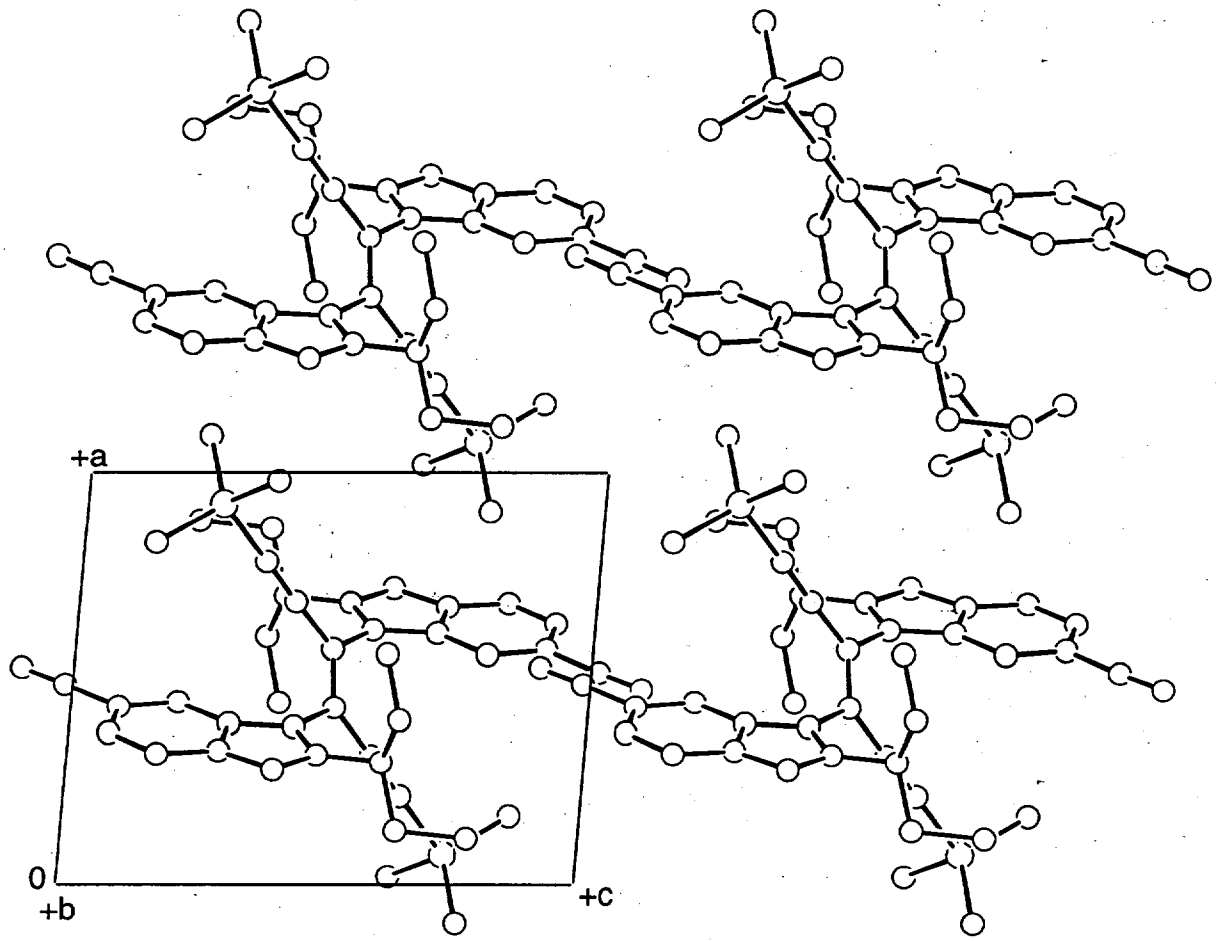
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Crystal structural analysis

Crystals were used as supplied. A crystal of dimensions 0.14 × 0.18 × 0.37 mm was mounted on a fiber. The orientation parameters and cell dimensions were obtained from the setting angles of an Enraf-Nonius CAD-4 diffractometer for 25 centered reflections in the range $9.1° \leq \theta \leq 11.8°$. Table 1 contains a summary of crystal data and the final residuals. A more extensive table including particulars of data collection and structure refinement is in the supplementary material. The crystal diffracted weakly and data collection was halted at θ 25°. The centric distribution of intensities indicated the space-group P(-1). Absorption corrections were not deemed necessary. A *SIR92* E-map[1] showed all the non-hydrogen atoms of the asymmetric unit (half the centrosymmetric molecule. The $SiMe_3$ group had two rotational positions; the occupany factors of major and minor carbon atoms were refined to 0.84(1), 0.16(1) and then fixed. Aromatic and ethyl hydrogen atoms were located and refined; the hydrogens on the major $SiMe_3$ carbons were included at positions recalculated after each cycle of refinement [$B(H) = 1.2B_{eq}(C)$; d(C-H) = 0.95 Å]. The final difference synthesis was featureless. The *TEXSAN* program suite,[2] incorporating complex atomic scattering factors, was used in all calculations.

References

1. Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.C.; Polidori, G.; Camalli, N. *J. Appl. Crystallography*, 1994, *27*, 435.

2. Molecular Structures Corporation, 3200A Research Forest Drive, The Woodlands, TX 77381, USA. *TeXsan Software for Single-Crystal Structure Analysis*, version 1.7, 1997.

Table 1

Crystallographic data

Composition	$C_{36}H_{44}N_8Si_2$	Z	1
Formula wt	644.97	d_{calc}	1.065 g cm^{-3}
Space-group	P(-1)	T	22 °C
a	9.2640(10) Å	λ	0.71073 Å
b	10.3494(11) Å	μ	1.21 cm^{-1}
c	11.3182(12) Å	No. obs. rflns	2128 [I≥σ(I)]
α	76.972(9)°	Total indep. rflns	3524
β	81.752(9)°	R(F), wR(F) [I≥σ(I)]	0.061, 0.058
γ	72.543(9)°	$R(F^2)$, $wR(F^2)$ (all)	0.085, 0.119
V	1005.2(9) $Å^3$		

$$R(F) = \Sigma||F_o|-|F_c||/\Sigma|F_o| \qquad wR(F^2) = [\Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w|F_o|^4]^{1/2}$$

Atomic coordinates and equivalent isotropic thermal parameters ($Å^2$)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a^*_i a^*_j a_i \cdot a_j$$

Atom	x	y	z	B_{eq}
Si	0.9256(1)	0.6334(1)	0.25990(9)	7.81(3)
N(1)	0.7202(3)	0.0865(2)	0.6022(2)	5.70(6)
N(2)	0.6850(2)	0.2068(2)	0.5202(2)	4.54(5)
N(3)	0.7023(3)	0.2109(2)	0.3941(2)	5.55(6)
N(4)	0.4730(5)	0.3563(4)	1.1004(3)	12.4(1)
C(1)	0.6103(3)	0.2723(2)	0.6937(2)	3.80(6)
C(2)	0.5583(3)	0.3357(3)	0.7946(2)	4.62(7)
C(3)	0.5696(3)	0.2531(3)	0.9082(2)	5.81(8)
C(4)	0.6322(4)	0.1076(4)	0.9230(3)	7.0(1)
C(5)	0.6826(4)	0.0450(4)	0.8276(3)	6.43(9)
C(6)	0.6746(3)	0.1286(3)	0.7097(2)	4.80(7)
C(7)	0.6181(2)	0.3204(2)	0.5686(2)	3.59(6)
C(8)	0.5718(2)	0.4597(2)	0.4938(2)	3.38(5)
C(9)	0.6873(3)	0.5092(2)	0.4152(2)	3.87(6)
C(10)	0.7839(3)	0.5536(3)	0.3524(2)	4.98(7)
C(11)	0.5145(4)	0.3119(4)	1.0147(3)	8.3(1)
C(12)	0.9802(6)	0.7316(6)	0.3621(6)	12.4(2)
C(12a)	0.902(3)	0.601(3)	0.092(2)	8.7(6)
C(13)	0.8318(8)	0.7612(10)	0.1373(6)	21.9(3)
C(13a)	1.103(5)	0.523(4)	0.296(3)	10.8(9)
C(14)	1.0927(8)	0.5024(9)	0.2237(9)	18.7(3)

C(14a)	0.890(3)	0.806(3)	0.264(3)	9.3(6)
C(15)	0.8667(5)	0.1593(5)	0.3560(4)	8.4(1)
C(16)	0.8856(7)	0.2006(8)	0.2165(5)	13.1(2)
C(17)	0.6026(6)	0.1349(4)	0.3652(3)	7.3(1)
C(18)	0.4406(6)	0.1991(5)	0.3991(4)	7.9(1)

Occupancy factors: C(12,13,14), 0.84(1); C(12a,13a,14a), 0.16(1)

Bond lengths (Å)

Si-C(10)	1.835(3)	C(1)-C(2)	1.400(3)
Si-C(12)	1.905(6)	C(1)-C(6)	1.407(3)
Si-C(13)	1.805(5)	C(1)-C(7)	1.390(3)
Si-C(14)	1.792(7)	C(2)-C(3)	1.373(4)
Si-C(12a)	2.05(2)	C(3)-C(4)	1.422(4)
Si-C(13a)	1.74(4)	C(3)-C(11)	1.432(5)
Si-C(14a)	1.73(3)	C(4)-C(5)	1.340(5)
N(1)-N(2)	1.356(3)	C(5)-C(6)	1.415(4)
N(1)-C(6)	1.353(3)	C(7)-C(8)	1.470(3)
N(2)-N(3)	1.406(3)	C(8)-C(8i)	1.348(4)
N(2)-C(7)	1.350(3)	C(8)-C(9)	1.432(3)
N(3)-C(15)	1.490(4)	C(9)-C(10)	1.198(3)
N(3)-C(17)	1.489(4)	C(15)-C(16)	1.539(7)
N(4)-C(11)	1.136(4)	C(17)-C(18)	1.481(5)

Bond angles (°)

C(10)-Si-C(12)	105.5(2)	C(6)-C(1)-C(7)	105.5(2)
C(10)-Si-C(13)	107.7(2)	C(1)-C(2)-C(3)	117.9(3)
C(10)-Si-C(14)	110.0(3)	C(2)-C(3)-C(4)	121.1(3)
C(10)-Si-C(12a)	99.7(7)	C(2)-C(3)-C(11)	120.4(3)
C(10)-Si-C(13a)	107(1)	C(4)-C(3)-C(11)	118.5(3)
C(10)-Si-C(14a)	113.5(9)	C(3)-C(4)-C(5)	121.9(3)
C(12)-Si-C(13)	106.6(4)	C(4)-C(5)-C(6)	117.9(3)
C(12)-Si-C(14)	107.4(4)	N(1)-C(6)-C(1)	111.9(2)
C(13)-Si-C(14)	118.8(5)	N(1)-C(6)-C(5)	127.3(3)
C(12a)-Si-C(13a)	104(1)	C(1)-C(6)-C(5)	120.8(3)
C(12a)-Si-C(13a)	113(1)	N(2)-C(7)-C(1)	104.9(2)
C(13a)-Si-C(14a)	118(2)	N(2)-C(7)-C(8)	122.8(2)
N(2)-N(1)-C(6)	102.6(2)	C(1)-C(7)-C(8)	132.3(2)
N(1)-N(2)-N(3)	122.2(2)	C(7)-C(8)-C(8)	120.7(2)
N(1)-N(2)-C(7)	115.1(2)	C(7)-C(8)-C(9)	117.3(2)
N(3)-N(2)-C(7)	122.4(2)	C(8)-C(8)-C(9)	121.8(2)
N(2)-N(3)-C(15)	108.8(2)	C(8)-C(9)-C(10)	177.6(3)
N(2)-N(3)-C(17)	108.6(2)	Si-C(10)-C(9)	175.9(2)
C(15)-N(3)-C(17)	115.5(3)	N(4)-C(11)-C(3)	178.6(4)
C(2)-C(1)-C(6)	120.4(2)	N(3)-C(15)-C(16)	107.2(4)
C(2)-C(1)-C(7)	134.1(2)	N(3)-C(17)-C(18)	111.6(3)

Supplementary material

Crystallographic information

Crystal data

Formula	$C_{36}H_{44}N_8Si_2$
Formula weight	644.97
Crystal system	triclinic
Space-group	P(-1)
a, b, c	9.2640(10), 10.3494(11), 11.3182(12) Å
α, β, γ	76.972(9), 81.752(9), 72.543(9)°
V	1005.2(2) Å^3
Z	1
d_{calc}	1.065 g cm^{-3}
μ	1.21 cm^{-1}
F_{000}	344

Data collection

Crystal appearance	colorless prism
Crystal dimensions	0.14 × 0.18 × 0.37 mm
Diffractometer	Enraf-Nonius CAD-4
Radiation, wavelength	Mo Kα, 0.71073 Å
Monochromator	graphite
Temperature	22 °C
$2\theta_{max}$	50°
Index range *h*, *k*, *l*	0→10, -11→12, -13→13
Scan mode	ω-2θ

Scan speed (on ω)	$4.1°\ min^{-1}$
Scan width	$(0.83 + 0.35tan\theta)°$
Reference reflections	3, every 3600 s exposure
No. indep. rflns scanned	3524
R_{int} [on F^2 for $\pm(0kl)$]	0.018

Refinement

Absorption correction	none
Sec. extinction parameter (g)	$1.4(3) \times 10^{-6}$
No. rflns in refinement (N):	2128 [$I \geq \sigma(I)$]
	3524 (all indep. data)
No. parameters (V)	273
Function minimized	$\Sigma w(\|F_o\|-\|F_c\|)^2$
Weighting factor w	$1/\sigma^2(F)$
R(F), wR(F) [$I \geq \sigma(I)$]	0.061, 0.058
$R(F^2)$, $wR(F^2)$ (all data)	0.085, 0.119
S [$I \geq \sigma(I)$], S (all data)	1.76, 1.47
Max. Δ/σ, last cycle	0.08
Max., min. in final diff. map	0.35, -0.35 e $Å^{-3}$

$$R(F) = \Sigma||F_o|-|F_c||/\Sigma|F_o| \qquad wR(F^2) = [\Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w|F_o|^4]^{1/2}$$

$$S = [\Sigma w(|F_o|-|F_c|)^2/(N-V)]^{1/2} \qquad I_o(corr) = I_o(1+2gI_c)$$

Supporting data

Coordinates and thermal parameters ($Å^2$) for hydrogen atoms

[H(1-13), refined; H(14-22), calculated parameters]

H(1)	0.517(3)	0.433(3)	0.787(2)	5.1(6)
H(2)	0.634(4)	0.055(3)	0.997(3)	8.7(9)
H(3)	0.725(3)	-0.046(3)	0.827(2)	5.7(7)
H(4)	0.890(4)	0.068(3)	0.390(3)	8.4(9)
H(5)	0.941(3)	0.211(3)	0.395(2)	7.2(8)
H(6)	0.975(5)	0.194(5)	0.207(4)	11(1)
H(7)	0.88(2)	0.33(1)	0.19(1)	55(1)
H(8)	0.838(7)	0.132(6)	0.190(5)	20.8(7)
H(9)	0.636(3)	0.033(3)	0.406(3)	8.3(8)
H(10)	0.616(3)	0.140(3)	0.281(3)	6.4(7)
H(11)	0.399(4)	0.305(4)	0.362(3)	12(1)
H(12)	0.374(4)	0.157(4)	0.373(3)	11(1)
H(13)	0.412(4)	0.194(3)	0.497(3)	9.2(8)
H(14)	1.0413	0.6679	0.4222	15
H(15)	1.0355	0.7918	0.3144	15
H(16)	0.8909	0.7840	0.4006	15
H(17)	0.8921	0.8223	0.1029	26
H(18)	0.8183	0.7166	0.0766	26
H(19)	0.7356	0.8119	0.1679	26
H(20)	1.1135	0.4295	0.2924	22
H(21)	1.0769	0.4676	0.1572	22
H(22)	1.1763	0.5407	0.2023	22

Supporting data

Anisotropic thermal parameters ($Å^2$)

The temperature factor is given by: $T = \exp[-2\pi^2\Sigma_i\Sigma_j U_{ij}h_ih_ja^*_ia^*_j]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si	0.0598(6)	0.1129(8)	0.1059(8)	-0.0383(6)	0.0093(5)	0.0253(6)
N(1)	0.086(2)	0.041(1)	0.075(2)	-0.005(1)	-0.014(1)	0.006(1)
N(2)	0.068(1)	0.045(1)	0.054(1)	-0.011(1)	-0.005(1)	-0.004(1)
N(3)	0.087(2)	0.060(1)	0.057(2)	-0.013(1)	0.003(1)	-0.013(1)
N(4)	0.211(4)	0.169(3)	0.058(2)	-0.019(3)	0.002(2)	-0.013(2)
C(1)	0.043(1)	0.048(2)	0.049(2)	-0.012(1)	-0.006(1)	0.001(1)
C(2)	0.054(2)	0.062(2)	0.054(2)	-0.017(1)	-0.005(1)	0.001(2)
C(3)	0.071(2)	0.086(2)	0.053(2)	-0.019(2)	-0.007(1)	0.003(2)
C(4)	0.093(3)	0.096(3)	0.061(2)	-0.026(2)	-0.014(2)	0.022(2)
C(5)	0.088(2)	0.056(2)	0.081(3)	-0.009(2)	-0.018(2)	0.019(2)
C(6)	0.063(2)	0.052(2)	0.061(2)	-0.015(1)	-0.011(1)	0.006(1)
C(7)	0.040(1)	0.040(1)	0.052(2)	-0.010(1)	-0.003(1)	-0.002(1)
C(8)	0.045(1)	0.044(1)	0.039(1)	-0.016(1)	-0.003(1)	-0.002(1)
C(9)	0.046(1)	0.046(1)	0.048(1)	-0.011(1)	-0.004(1)	0.002(1)
C(10)	0.051(2)	0.066(2)	0.062(2)	-0.017(1)	-0.001(1)	0.008(1)
C(11)	0.126(3)	0.124(3)	0.047(2)	-0.021(2)	-0.003(2)	0.002(2)
C(12)	0.103(4)	0.138(4)	0.246(7)	-0.070(4)	-0.021(4)	-0.015(5)
C(13)	0.198(7)	0.39(1)	0.203(6)	-0.197(8)	-0.102(6)	0.227(8)
C(14)	0.121(5)	0.244(8)	0.36(1)	-0.082(5)	0.143(7)	-0.148(9)
C(15)	0.102(3)	0.100(3)	0.088(3)	0.005(3)	0.019(2)	-0.022(2)
C(16)	0.136(5)	0.231(7)	0.116(4)	-0.041(5)	0.050(4)	-0.055(4)
C(17)	0.154(4)	0.062(2)	0.067(2)	-0.036(2)	-0.016(2)	-0.011(2)
C(18)	0.115(4)	0.112(3)	0.094(3)	-0.062(3)	-0.021(3)	-0.012(3)

Supporting data

Refined C-H bond lengths (Å)

C(2)	H(1)	0.95(2)	C(16)	H(8)	1.05(6)
C(4)	H(2)	0.89(3)	C(17)	H(9)	1.02(3)
C(5)	H(3)	0.91(3)	C(17)	H(10)	0.93(3)
C(15)	H(4)	0.91(3)	C(18)	H(11)	1.05(4)
C(15)	H(5)	1.16(3)	C(18)	H(12)	0.96(4)
C(16)	H(6)	0.80(4)	C(18)	H(13)	1.09(3)
C(16)	H(7)	1.3(1)			

Supporting data

Intermolecular distances (Å) for non-hydrogen atoms

N(4)	C(11^{i})	3.418(5)	N(4)	C(2^{i})	3.564(5)
N(4)	C(18^{ii})	3.418(5)	N(4)	C(14^{iii})	3.604(7)
N(4)	N(4^{i})	3.433(7)	C(19	C(19^{iv})	3.26(5)
N(4)	C(17^{ii})	3.458(5)			

Symmmetry code: (i) 1-x, 1-y, 2-z; (ii) x, y, 1+z;

(iii) -1+x, y, 1+z; (iv) 2-x, 1-y, -z

Supporting data

Torsion angles (°)

(1)	(2)	(3)	(4)	angle	(1)	(2)	(3)	(4)	angle
Si	C(10)	C(9)	C(8)	-33(8)	C(2)	C(1)	C(6)	C(5)	2.4(4)
N(1)	N(2)	N(3)	C(15)	-65.2(4)	C(2)	C(1)	C(7)	C(8)	-0.8(5)
N(1)	N(2)	N(3)	C(17)	61.4(3)	C(2)	C(3)	C(4)	C(5)	0.0(5)
N(1)	N(2)	C(7)	C(1)	1.1(3)	C(3)	C(2)	C(1)	C(6)	-1.2(4)
N(1)	N(2)	C(7)	C(8)	-179.9(2)	C(3)	C(2)	C(1)	C(7)	-179.6(3)
N(1)	C(6)	C(1)	C(2)	-179.1(2)	C(3)	C(4)	C(5)	C(6)	1.2(5)
N(1)	C(6)	C(1)	C(7)	-0.3(3)	C(5)	C(4)	C(3)	C(11)	178.3(3)
N(1)	C(6)	C(5)	C(4)	179.4(3)	C(5)	C(6)	C(1)	C(7)	-178.8(3)
N(2)	N(1)	C(6)	C(1)	0.9(3)	C(6)	N(1)	N(2)	C(7)	-1.3(3)
N(2)	N(1)	C(6)	C(5)	179.3(3)	C(6)	C(1)	C(7)	C(8)	-179.3(2)
N(2)	N(3)	C(15)	C(16)	-165.2(4)	C(7)	N(2)	N(3)	C(15)	121.8(3)
N(2)	N(3)	C(17)	C(18)	61.2(3)	C(7)	N(2)	N(3)	C(17)	-111.7(3)
N(2)	C(7)	C(1)	C(2)	178.1(3)	C(7)	C(8)	C(8)	C(7)	180
N(2)	C(7)	C(1)	C(6)	-0.5(3)	C(7)	C(8)	C(8)	C(9)	-4.8(5)
N(2)	C(7)	C(8)	C(8)	126.2(3)	C(7)	C(8)	C(9)	C(10)	-104(6)
N(2)	C(7)	C(8)	C(9)	-58.4(3)	C(8)	C(8)	C(9)	C(10)	-72(6)
N(3)	N(2)	N(1)	C(6)	-174.8(2)	C(9)	C(8)	C(8)	C(9)	180
N(3)	N(2)	C(7)	C(1)	174.6(2)	C(9)	C(10)	Si	C(12)	44(3)
N(3)	N(2)	C(7)	C(8)	-6.4(3)	C(9)	C(10)	Si	C(13)	-70(3)
N(4)	C(11)	C(3)	C(2)	-151(18)	C(9)	C(10)	Si	C(14)	159(3)
N(4)	C(11)	C(3)	C(4)	30(19)	C(9)	C(10)	Si	C(12a)	-125(3)
C(1)	C(2)	C(3)	C(4)	0.0(4)	C(9)	C(10)	Si	C(13a)	127(3)
C(1)	C(2)	C(3)	C(11)	-178.3(3)	C(9)	C(10)	Si	C(14a)	-4(4)

C(1)	C(6)	C(5)	C(4)	-2.4(5)	C(15)	N(3)	C(17)	C(18)	-176.3(3)
C(1)	C(7)	C(8)	C(8)	-55.1(4)	C(16)	C(15)	N(3)	C(17)	72.3(5)
C(1)	C(7)	C(8)	C(9)	120.2(3)					

The sign is positive if a clockwise motion of atom 1 would superimpose it on atom 4 when the direction of view is from atom 2 to atom 3.

Supporting data

Least-squares mean planes (deviations in Å)

Plane 1	Defining atoms	Distance	Other atoms	Distance
	C(1)	-0.007(2)	N(1)	0.020
	C(2)	0.002(3)	C(7)	-0.004
	C(3)	0.004(3)	C(11)	-0.023
	C(4)	-0.002(4)	H(1)	0.013
	C(5)	-0.011(4)	H(2)	-0.046
	C(6)	0.012(3)	H(3)	-0.007

Mean deviation from plane: 0.006 Å; χ^2, 44.3

Plane 2	Defining atoms	Distance	Other atoms	Distance
	N(1)	-0.007(3)	N(3)	-0.107
	N(2)	0.006(2)	C(2)	0.030
	C(1)	0.001(2)	C(5)	-0.016
	C(6)	0.004(3)	C(8)	0.003
	C(7)	-0.005(2)		

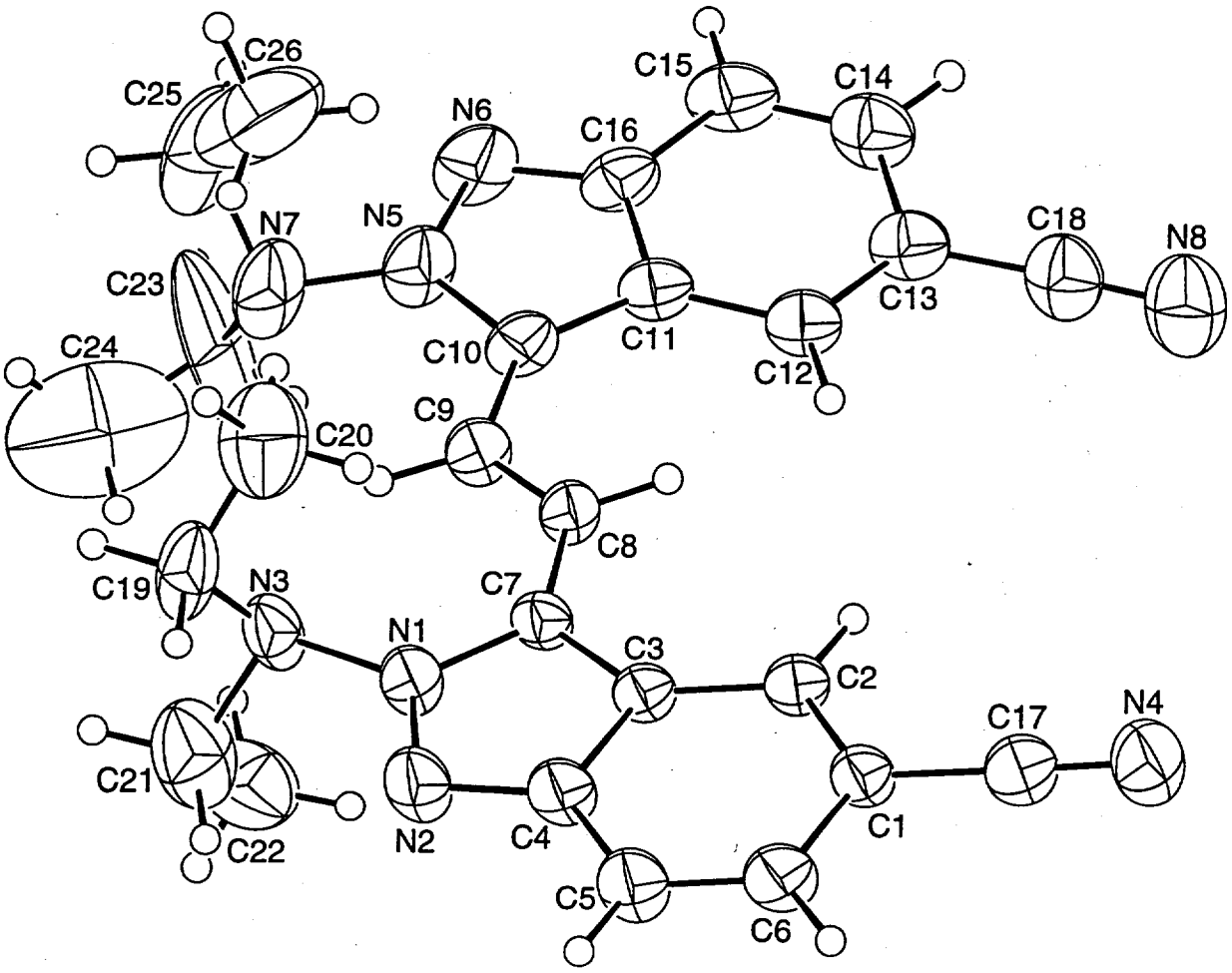
Mean deviation from plane: 0.005 Å; χ^2, 20.9

Plane 3	Defining atoms	Distance	Other atoms	Distance
	Si	0.008(1)	C(7)	0.314
	C(8)	0.041(3)		
	C(9)	-0.029(3)		
	C(10)	-0.043(3)		
	Si^{i}	-0.008(1)		
	$C(8^{i})$	-0.041(3)		
	$C(9^{i})$	0.029(3)		
	$C(10^{i})$	0.043(3)		

Mean deviation from plane: 0.030 Å; χ^2, 712

Dihedral angles between planes

2	1	0.9°
3	1	58.2
3	2	57.5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C25
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C14
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C16
N5
N7
C18
N8
C23
C13
C24
C10
C11
C12
C9
C20
C8
C19
N3
C7
N1
C3
C2
C17
N4
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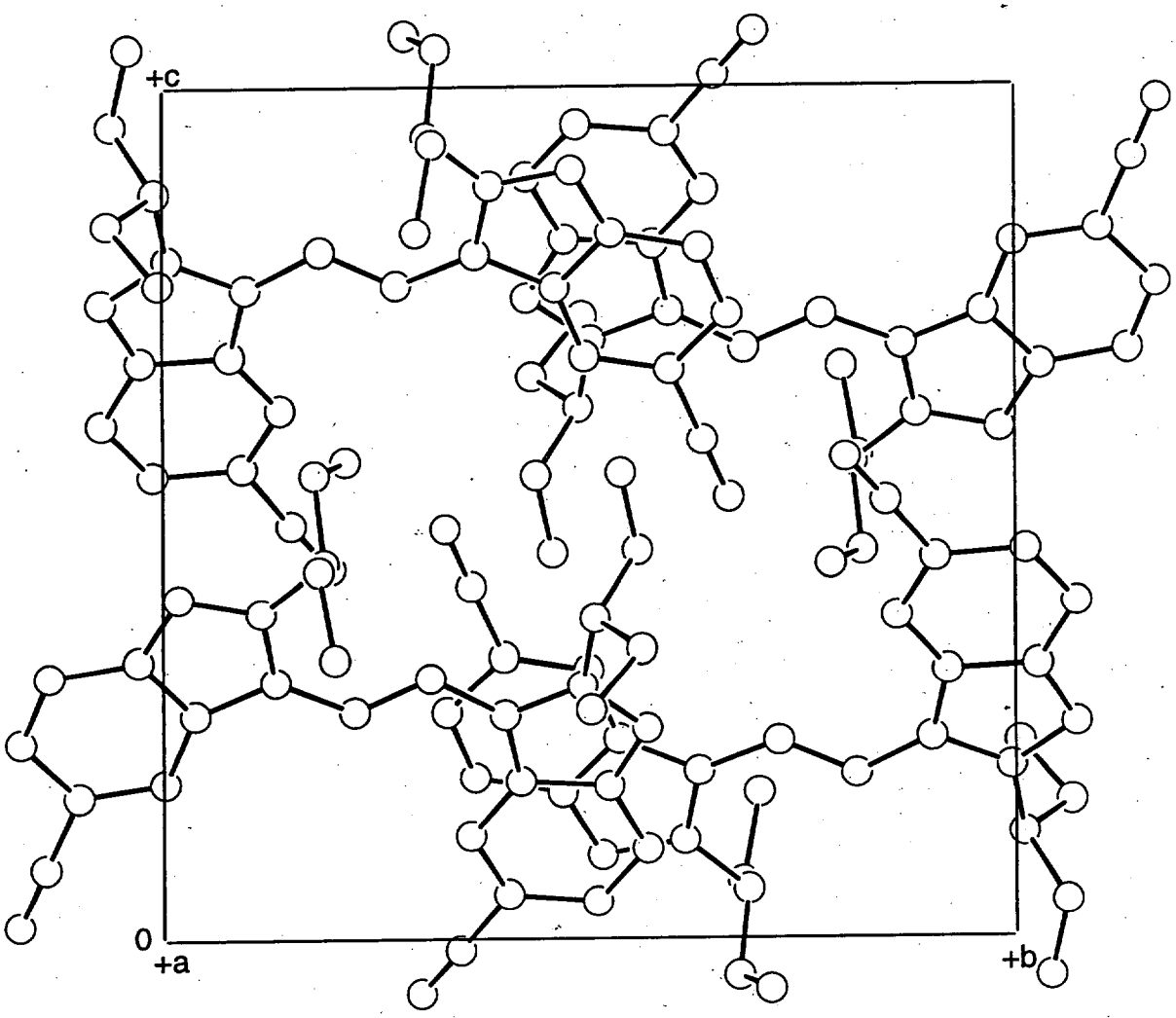
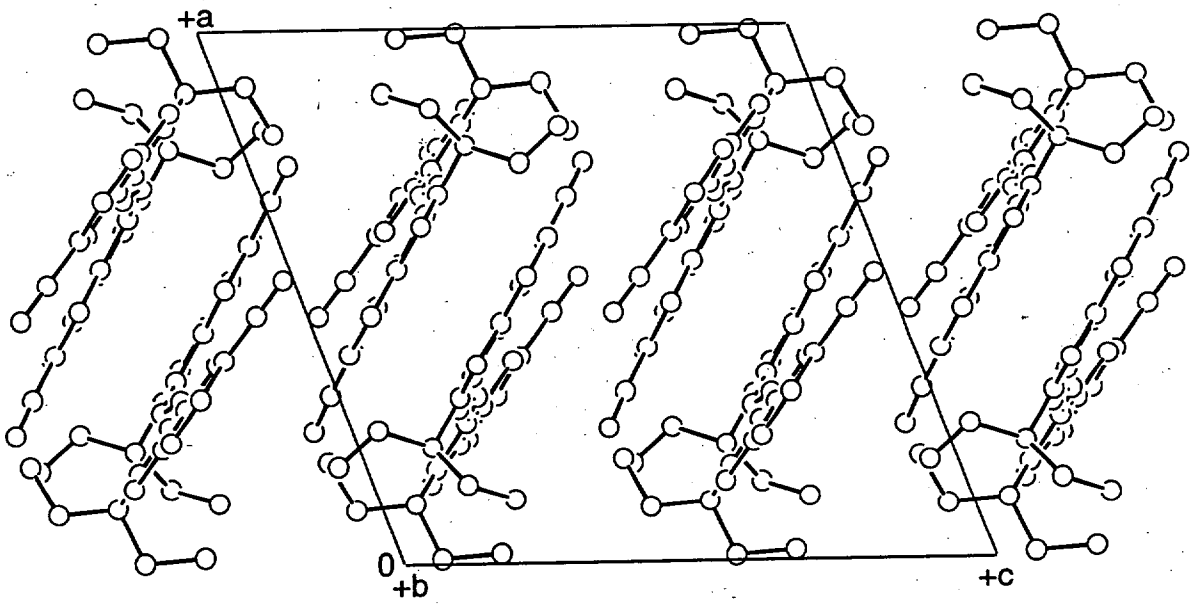
C1
N2
C4
C22
C5
C6


+c
0
+a
+b


+a
0
+b
+c

Crystal structural analysis

A yellow tablet of dimensions 0.05 × 0.43 × 0.43 mm was mounted on a fiber. The orientation parameters and cell dimensions were obtained from the setting angles of an Enraf-Nonius CAD-4 diffractometer for 25 centered reflections in the range $9.1° \leq \theta \leq 12.0°$. Table 1 contains a summary of crystal data and the final residuals. A more extensive table including particulars of data collection and structure refinement is in the supplementary material. The crystal diffracted weakly and data collection was halted at θ 22.5° at which point almost all reflections had intensities less than $\sigma(I)$. The systematic absences indicated the space-group $P2_1/c$. Absorption corrections based on azimuthal scans ('ψ-scans') were applied. A *SIR92* *E*-map[1] showed all the non-hydrogen atoms. Hydrogen atoms were included at positions recalculated after each cycle of refinement [$B(H) = 1.2B_{eq}(C)$; d(C-H) = 0.95 Å]. The final difference synthesis was featureless. The *TEXSAN* program suite,[2] incorporating complex atomic scattering factors, was used in all calculations.

References

1. Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.C.; Polidori, G.; Camalli, N. *J. Appl. Crystallography*, 1994, *27*, 435.

2. Molecular Structures Corporation, 3200A Research Forest Drive, The Woodlands, TX 77381, USA. *TeXsan Software for Single-Crystal Structure Analysis*, version 1.7, 1997.

Table 1

Crystallographic data

Composition	$C_{28}H_{26}N_8$	d_{calc}	1.187 g cm^{-3}
Formula wt	452.6	T	22 °C
Space-group	$P2_1/c$	λ	0.71073 Å
a	13.899(2) Å	μ	0.75 cm^{-1}
b	13.495(2) Å	No. obs. rflns	1839 [$I \geq \sigma(I)$]
c	14.436(3) Å	Total indep. rflns	3298
β	110.71(2)°	R(F), wR(F)	0.078, 0.072
V	2533(1) $Å^3$	$R(F^2)$, $wR(F^2)$	0.116, 0.146
Z	4		

$$R(F) = \Sigma||F_o|-|F_c||/\Sigma|F_o| \qquad wR(F^2) = [\Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w|F_o|^4]^{1/2}$$

Atomic coordinates and equivalent isotropic thermal parameters ($Å^2$)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a^*_i a^*_j a_i \cdot a_j$$

Atom	x	y	z	B_{eq}
N(1)	0.8222(3)	0.1149(2)	0.3834(3)	5.4(1)
N(2)	0.8502(3)	0.0179(3)	0.4012(3)	6.4(1)
N(3)	0.8869(4)	0.1892(3)	0.4411(3)	6.3(1)
N(4)	0.4660(4)	-0.1675(3)	0.0181(3)	8.4(1)
N(5)	0.6898(3)	0.4948(3)	0.2974(3)	6.6(1)
N(6)	0.6297(3)	0.5712(3)	0.2476(4)	7.1(1)
N(7)	0.7816(4)	0.5096(3)	0.3761(4)	8.8(1)
N(8)	0.2515(4)	0.3028(4)	-0.0644(4)	9.5(2)
C(1)	0.6150(3)	-0.0981(3)	0.1689(3)	4.8(1)
C(2)	0.6228(3)	0.0020(3)	0.1840(3)	4.7(1)
C(3)	0.7048(3)	0.0372(3)	0.2644(3)	4.3(1)
C(4)	0.7771(4)	-0.0292(3)	0.3280(3)	5.3(1)
C(5)	0.7664(4)	-0.1325(3)	0.3104(4)	6.3(1)
C(6)	0.6866(4)	-0.1659(3)	0.2332(4)	5.8(1)
C(7)	0.7369(3)	0.1317(3)	0.3029(3)	4.4(1)
C(8)	0.6883(3)	0.2249(3)	0.2673(3)	4.6(1)
C(9)	0.7097(3)	0.3149(3)	0.3065(3)	5.0(1)
C(10)	0.6528(4)	0.4029(3)	0.2632(4)	5.2(1)
C(11)	0.5608(4)	0.4213(3)	0.1851(4)	5.0(1)
C(12)	0.4822(4)	0.3622(3)	0.1218(3)	5.3(1)
C(13)	0.4000(4)	0.4070(4)	0.0530(4)	5.8(1)
C(14)	0.3932(4)	0.5119(4)	0.0446(4)	6.9(2)
C(15)	0.4654(4)	0.5712(3)	0.1061(4)	6.6(2)
C(16)	0.5509(4)	0.5260(3)	0.1793(4)	5.7(1)
C(17)	0.5314(4)	-0.1368(3)	0.0857(4)	6.1(1)
C(18)	0.3171(4)	0.3494(4)	-0.0135(4)	7.1(2)
C(19)	0.9906(4)	0.1829(4)	0.4324(5)	8.5(2)
C(20)	0.9819(5)	0.2017(5)	0.3285(6)	11.2(2)
C(21)	0.8966(6)	0.1780(5)	0.5444(6)	10.7(2)
C(22)	0.8072(7)	0.2144(6)	0.5603(6)	13.6(3)

C(23)	0.7482(9)	0.5602(7)	0.4564(7)	20.7(4)
C(24)	0.815(1)	0.5404(8)	0.547(1)	23.7(6)
C(25)	0.8518(7)	0.5650(5)	0.3398(9)	14.6(3)
C(26)	0.8798(6)	0.5050(7)	0.2692(9)	17.1(4)

Bond lengths (Å)

N(1)-N(2)	1.363(4)	C(3)-C(7)	1.400(5)
N(1)-N(3)	1.407(4)	C(4)-C(5)	1.415(6)
N(1)-C(7)	1.354(4)	C(5)-C(6)	1.342(5)
N(2)-C(4)	1.340(5)	C(7)-C(8)	1.434(5)
N(3)-C(19)	1.493(7)	C(8)-C(9)	1.328(5)
N(3)-C(21)	1.456(8)	C(9)-C(10)	1.441(5)
N(4)-C(17)	1.150(5)	C(10)-C(11)	1.396(5)
N(5)-N(6)	1.361(5)	C(11)-C(12)	1.400(6)
N(5)-N(7)	1.390(5)	C(11)-C(16)	1.419(5)
N(5)-C(10)	1.366(5)	C(12)-C(13)	1.362(6)
N(6)-C(16)	1.334(5)	C(13)-C(14)	1.421(6)
N(7)-C(23)	1.551(12)	C(13)-C(18)	1.439(7)
N(7)-C(25)	1.466(12)	C(14)-C(15)	1.344(6)
N(8)-C(18)	1.137(6)	C(15)-C(16)	1.419(6)
C(1)-C(2)	1.366(5)	C(19)-C(20)	1.485(8)
C(1)-C(6)	1.427(5)	C(21)-C(22)	1.428(9)
C(1)-C(17)	1.442(6)	C(23)-C(24)	1.334(16)
C(2)-C(3)	1.391(5)	C(25)-C(26)	1.458(13)
C(3)-C(4)	1.415(5)		

Bond angles (°)

N(2)-N(1)-N(3)	119.5(3)	N(1)-C(7)-C(3)	104.3(3)
N(2)-N(1)-C(7)	115.4(3)	N(1)-C(7)-C(8)	127.9(4)
N(3)-N(1)-C(7)	124.8(3)	C(3)-C(7)-C(8)	127.8(4)
N(1)-N(2)-C(4)	102.7(3)	C(7)-C(8)-C(9)	130.0(4)
N(1)-N(3)-C(19)	110.1(4)	C(8)-C(9)-C(10)	124.3(4)
N(1)-N(3)-C(21)	109.9(4)	N(5)-C(10)-C(9)	120.8(4)
C(19)-N(3)-C(21)	109.7(5)	N(5)-C(10)-C(11)	104.5(4)
N(6)-N(5)-N(7)	122.5(4)	C(9)-C(10)-C(11)	134.6(4)
N(6)-N(5)-C(10)	114.4(4)	C(10)-C(11)-C(12)	134.9(4)
N(7)-N(5)-C(10)	123.1(4)	C(10)-C(11)-C(16)	105.5(4)
N(5)-N(6)-C(16)	103.6(4)	C(12)-C(11)-C(16)	119.5(4)
N(5)-N(7)-C(23)	104.1(5)	C(11)-C(12)-C(13)	118.9(4)
N(5)-N(7)-C(25)	108.4(6)	C(12)-C(13)-C(14)	121.2(5)
C(23)-N(7)-C(25)	117.1(7)	C(12)-C(13)-C(18)	120.9(4)
C(2)-C(1)-C(6)	122.1(4)	C(14)-C(13)-C(18)	117.9(5)
C(2)-C(1)-C(17)	119.1(4)	C(13)-C(14)-C(15)	121.7(5)
C(6)-C(1)-C(17)	118.8(4)	C(14)-C(15)-C(16)	117.9(4)
C(1)-C(2)-C(3)	117.8(4)	N(6)-C(16)-C(11)	112.0(4)
C(2)-C(3)-C(4)	120.6(4)	N(6)-C(16)-C(15)	127.3(4)
C(2)-C(3)-C(7)	134.0(4)	C(11)-C(16)-C(15)	120.7(5)
C(4)-C(3)-C(7)	105.4(4)	N(4)-C(17)-C(1)	178.6(6)
N(2)-C(4)-C(3)	112.2(3)	N(8)-C(18)-C(13)	178.6(7)
N(2)-C(4)-C(5)	127.7(4)	N(3)-C(19)-C(20)	109.9(5)
C(3)-C(4)-C(5)	120.1(4)	N(3)-C(21)-C(22)	111.0(7)
C(4)-C(5)-C(6)	118.9(4)	N(7)-C(23)-C(24)	110.5(9)
C(1)-C(6)-C(5)	120.5(4)	N(7)-C(25)-C(26)	110.0(7)

Supplementary material

Crystallographic information

Crystal data

Formula	$C_{28}H_{26}N_8$
Formula weight	452.6
Crystal system	monoclinic
Space-group	$P2_1/c$
a, b, c	13.899(2), 13.485(2), 14.436(3) Å
α, β, γ	90, 110.71(2), 90°
V	2533(1) Å^3
Z	4
d_{calc}	1.187 g cm^{-3}
μ	0.75 cm^{-1}
F_{000}	960

Data collection

Crystal appearance	yellow tablet
Crystal dimensions	0.05 × 0.43 × 0.43 mm
Diffractometer	Enraf-Nonius CAD-4
Radiation, wavelength	Mo Kα, 0.71073 Å
Monochromator	graphite
Temperature	22 °C
$2\theta_{max}$	45°
Index range *h*, *k*, *l*	0→14, 0→14, -14→14
Scan mode	ω-2θ

Scan speed (on ω)	4.1° min^{-1}
Scan width	(0.80 + 0.35tanθ)°
Reference reflections	3, every 3600 s exposure
No. indep. rflns scanned	3479 (incl. 181 syst. absent)
R_{int} (on F^2 for *0,k,±l*)	0.037

Refinement

Absorption correction	none
Sec. extinction parameter (g)	zero
No. rflns in refinement (N):	1839 [I≥σ(I)]
	3298 (all indep. data)
No. parameters (V)	307
Function minimized	$\Sigma w(\|F_o\|-\|F_c\|)^2$
Weighting factor w	$1/\sigma^2(F)$
R(F), wR(F) [I≥σ(I)]	0.078, 0.072
$R(F^2)$, $wR(F^2)$ (all data)	0.116, 0.146
S [I≥σ(I)], S (all data)	2.10, 1.68
Max. Δ/σ, last cycle	0.01
Max., min. in final diff. map	0.48, -0.39 e Å^{-3}

$$R(F) = \Sigma||F_o|-|F_c||/\Sigma|F_o| \qquad wR(F^2) = [\Sigma w(|F_o|^2-|F_c|^2)^2/\Sigma w|F_o|^4]^{1/2}$$

$$S = [\Sigma w(|F_o|-|F_c|)^2/(N-V)]^{1/2} \qquad I_o(corr) = I_o(1+2gI_c)$$

Supporting data

Anisotropic thermal parameters ($Å^2$)

The temperature factor is given by: $T = \exp[-2\pi^2\Sigma_i\Sigma_j U_{ij}h_ih_ja^*_ia^*_j]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	0.082(3)	0.039(2)	0.065(3)	0.004(2)	0.003(2)	-0.003(2)
N(2)	0.092(3)	0.045(2)	0.078(3)	0.011(2)	-0.005(2)	0.006(2)
N(3)	0.099(4)	0.053(2)	0.059(3)	-0.001(2)	-0.006(3)	-0.007(2)
N(4)	0.125(4)	0.060(3)	0.098(4)	-0.006(3)	-0.006(3)	-0.003(3)
N(5)	0.088(3)	0.045(2)	0.104(3)	0.002(2)	0.018(3)	-0.008(2)
N(6)	0.095(3)	0.036(2)	0.132(4)	0.014(2)	0.030(3)	0.005(2)
N(7)	0.110(4)	0.050(3)	0.139(5)	-0.008(3)	0.000(4)	-0.022(3)
N(8)	0.087(4)	0.131(5)	0.127(4)	0.013(3)	0.016(3)	-0.007(4)
C(1)	0.077(3)	0.042(3)	0.056(3)	0.001(2)	0.013(3)	0.002(2)
C(2)	0.068(3)	0.039(3)	0.065(3)	0.004(2)	0.014(3)	0.006(2)
C(3)	0.068(3)	0.032(2)	0.059(3)	0.004(2)	0.016(3)	0.003(2)
C(4)	0.084(3)	0.035(3)	0.068(3)	0.011(2)	0.011(3)	0.003(2)
C(5)	0.094(4)	0.043(3)	0.084(4)	0.013(3)	0.007(3)	0.005(3)
C(6)	0.101(4)	0.036(3)	0.075(4)	0.005(3)	0.019(3)	-0.001(3)
C(7)	0.064(3)	0.042(3)	0.054(3)	0.004(2)	0.011(3)	0.007(2)
C(8)	0.063(3)	0.039(3)	0.063(3)	0.002(2)	0.010(2)	-0.003(2)
C(9)	0.073(3)	0.043(3)	0.067(3)	0.004(2)	0.016(3)	-0.003(2)
C(10)	0.080(3)	0.033(3)	0.083(4)	0.001(2)	0.030(3)	-0.003(3)
C(11)	0.071(3)	0.044(3)	0.078(4)	0.013(3)	0.032(3)	0.008(3)
C(12)	0.074(3)	0.051(3)	0.079(4)	0.014(3)	0.030(3)	0.007(3)
C(13)	0.070(4)	0.069(3)	0.088(4)	0.016(3)	0.035(3)	0.013(3)
C(14)	0.082(4)	0.083(4)	0.096(4)	0.024(3)	0.030(3)	0.020(3)
C(15)	0.089(4)	0.053(3)	0.117(5)	0.031(3)	0.047(4)	0.028(3)
C(16)	0.077(3)	0.045(3)	0.100(4)	0.004(3)	0.038(3)	0.011(3)
C(17)	0.095(4)	0.042(3)	0.083(4)	0.001(3)	0.015(3)	0.002(3)
C(18)	0.076(4)	0.084(4)	0.099(5)	0.018(3)	0.021(4)	0.002(3)
C(19)	0.060(4)	0.073(4)	0.145(6)	0.004(3)	-0.018(4)	-0.005(4)
C(20)	0.082(5)	0.175(7)	0.160(7)	0.013(4)	0.034(5)	-0.017(6)
C(21)	0.154(7)	0.112(5)	0.111(6)	0.008(5)	0.012(6)	-0.032(4)

C(22)	0.23(1)	0.180(8)	0.097(6)	-0.046(7)	0.045(6)	0.008(6)
C(23)	0.29(1)	0.203(9)	0.144(8)	0.140(8)	-0.111(8)	-0.146(7)
C(24)	0.47(2)	0.170(9)	0.37(2)	0.02(1)	0.28(2)	-0.05(1)
C(25)	0.127(7)	0.060(5)	0.29(1)	-0.002(4)	-0.015(7)	-0.016(6)
C(26)	0.135(7)	0.163(8)	0.35(1)	-0.013(7)	0.087(8)	0.097(9)

Supporting data

Calculated coordinates and thermal parameters ($Å^2$) of hydrogen atoms

Atom	x	y	z	B
H(1)	0.5740	0.0461	0.1410	5.7
H(2)	0.8150	-0.1774	0.3524	7.6
H(3)	0.6777	-0.2351	0.2214	7.0
H(4)	0.6322	0.2218	0.2061	5.5
H(5)	0.7661	0.3216	0.3671	6.0
H(6)	0.4862	0.2920	0.1267	6.4
H(7)	0.3361	0.5408	-0.0057	8.3
H(8)	0.4595	0.6413	0.1006	7.9
H(9)	1.0347	0.2311	0.4744	10.2
H(10)	1.0184	0.1187	0.4518	10.2
H(11)	0.9537	0.2657	0.3089	13.4
H(12)	1.0482	0.1981	0.3235	13.4
H(13)	0.9384	0.1532	0.2865	13.4
H(14)	0.9051	0.1098	0.5617	12.8
H(15)	0.9550	0.2140	0.5849	12.8
H(16)	0.7998	0.2833	0.5458	16.4
H(17)	0.7482	0.1802	0.5182	16.4
H(18)	0.8141	0.2041	0.6274	16.4
H(19)	0.7449	0.6298	0.4461	24.9
H(20)	0.6823	0.5362	0.4515	24.9
H(21)	0.8199	0.4707	0.5567	28.5
H(22)	0.8811	0.5656	0.5523	28.5
H(23)	0.7932	0.5707	0.5949	28.5
H(24)	0.8189	0.6240	0.3081	17.5
H(25)	0.9121	0.5817	0.3940	17.5
H(26)	0.8195	0.4876	0.2153	20.5
H(27)	0.9245	0.5418	0.2454	20.5
H(28)	0.9137	0.4465	0.3010	20.6

Supporting data

Intermolecular distances (Å) for non-hydrogen atoms

atom	atom	distance	code	atom	atom	distance	code
N(4)	C(2)	3.532(6)	6553	C(4)	C(14)	3.517(7)	6452
N(4)	C(12)	3.539(6)	6553	C(4)	C(13)	3.569(7)	6452
N(8)	C(6)	3.403(7)	6553	C(5)	C(18)	3.523(8)	6452
N(8)	C(20)	3.512(8)	4544	C(5)	C(13)	3.570(7)	6452
N(8)	C(19)	3.615(9)	4544	C(7)	C(15)	3.593(7)	6452
N(8)	C(23)	3.625(9)	6452	C(13)	C(15)	3.449(7)	6653
C(2)	C(16)	3.633(7)	6452	C(14)	C(15)	3.588(7)	6653
C(3)	C(14)	3.497(7)	6452	C(14)	C(16)	3.619(8)	6653
C(3)	C(15)	3.527(7)	6452				

The digits in the code pqrs denote that the second atom is derived from the corresponding atom in the table of atomic coordinates by the action of the symmetry operator numbered s (see below) followed by (p-5), (q-5), and (r-5) unit translations in the +a, +b, and +c directions respectively. For instance, the code 6452 denotes that the second atom has been shifted from x, y, z to 1-x, -0.5+y, 0.5-z.

(1)	+x,	+y,	+z	(2)	-x,	0.5+y,	0.5-z
(3)	-x,	-y,	-z	(4)	+x,	0.5-y,	0.5+z

Supporting data

Torsion angles (°)

(1)	(2)	(3)	(4)	angle	(1)	(2)	(3)	(4)	angle
N(1)	N(2)	C(4)	C(3)	0.6(5)	C(1)	C(2)	C(3)	C(4)	-0.1(7)
N(1)	N(2)	C(4)	C(5)	-179.0(5)	C(1)	C(2)	C(3)	C(7)	-179.3(5)
N(1)	N(3)	C(19)	C(20)	63.0(5)	C(1)	C(6)	C(5)	C(4)	1.3(8)
N(1)	N(3)	C(21)	C(22)	-77.5(7)	C(2)	C(1)	C(6)	C(5)	-1.6(8)
N(1)	C(7)	C(3)	C(2)	179.2(5)	C(2)	C(3)	C(4)	C(5)	-0.1(7)
N(1)	C(7)	C(3)	C(4)	-0.1(5)	C(2)	C(3)	C(7)	C(8)	1.1(9)
N(1)	C(7)	C(8)	C(9)	-5.1(8)	C(3)	C(2)	C(1)	C(6)	0.9(7)
N(2)	N(1)	N(3)	C(19)	58.5(6)	C(3)	C(2)	C(1)	C(17)	-179.4(4)
N(2)	N(1)	N(3)	C(21)	-62.5(6)	C(3)	C(4)	C(5)	C(6)	-0.5(8)
N(2)	N(1)	C(7)	C(3)	0.5(5)	C(3)	C(7)	C(8)	C(9)	172.5(5)
N(2)	N(1)	C(7)	C(8)	178.6(4)	C(4)	N(2)	N(1)	C(7)	-0.7(5)
N(2)	C(4)	C(3)	C(2)	-179.7(4)	C(4)	C(3)	C(7)	C(8)	-178.2(5)
N(2)	C(4)	C(3)	C(7)	-0.3(6)	C(5)	C(4)	C(3)	C(7)	179.3(5)
N(2)	C(4)	C(5)	C(6)	179.1(5)	C(5)	C(6)	C(1)	C(17)	178.8(5)
N(3)	N(1)	N(2)	C(4)	-174.4(4)	C(7)	N(1)	N(3)	C(19)	-114.6(5)
N(3)	N(1)	C(7)	C(3)	173.8(4)	C(7)	N(1)	N(3)	C(21)	124.5(5)
N(3)	N(1)	C(7)	C(8)	-8.1(7)	C(7)	C(8)	C(9)	C(10)	-178.8(5)
N(4)	C(17)	C(1)	C(2)	89(19)	C(8)	C(9)	C(10)	C(11)	9.8(9)
N(4)	C(17)	C(1)	C(6)	-92(19)	C(9)	C(10)	C(11)	C(12)	6(1)
N(5)	N(6)	C(16)	C(11)	-0.3(6)	C(9)	C(10)	C(11)	C(16)	-177.9(5)
N(5)	N(6)	C(16)	C(15)	-179.2(5)	C(10)	N(5)	N(6)	C(16)	0.4(5)
N(5)	N(7)	C(23)	C(24)	153(1)	C(10)	N(5)	N(7)	C(23)	-116.5(7)
N(5)	N(7)	C(25)	C(26)	-65.8(8)	C(10)	N(5)	N(7)	C(25)	118.1(6)

N(5) C(10)C(9) C(8)	-168.0(5)	C(10)C(11)C(12)C(13)	178.6(5)
N(5) C(10)C(11)C(12)	-176.0(5)	C(10)C(11)C(16)C(15)	179.1(5)
N(5) C(10)C(11)C(16)	0.1(5)	C(11)C(12)C(13)C(14)	0.2(7)
N(6) N(5) N(7) C(23)	64.0(8)	C(11)C(12)C(13)C(18)	-179.8(5)
N(6) N(5) N(7) C(25)	-61.4(7)	C(11)C(16)C(15)C(14)	1.9(8)
N(6) N(5) C(10)C(9)	178.1(4)	C(12)C(11)C(16)C(15)	-4.0(8)
N(6) N(5) C(10)C(11)	-0.3(5)	C(12)C(13)C(14)C(15)	-2.4(9)
N(6) C(16)C(11)C(10)	0.1(6)	C(13)C(12)C(11)C(16)	2.8(7)
N(6) C(16)C(11)C(12)	177.0(4)	C(13)C(14)C(15)C(16)	1.2(8)
N(6) C(16)C(15)C(14)	-179.2(5)	C(15)C(14)C(13)C(18)	177.6(5)
N(7) N(5) N(6) C(16)	180.0(5)	C(19)N(3) C(21)C(22)	161.3(6)
N(7) N(5) C(10)C(9)	-1.5(7)	C(20)C(19)N(3) C(21)	-175.9(5)
N(7) N(5) C(10)C(11)	-179.9(5)	C(23)N(7) C(25)C(26)	176.8(7)
N(8) C(18)C(13)C(12)	51(24)	C(24)C(23)N(7) C(25)	-88(1)
N(8) C(18)C(13)C(14)	-129(24)		

The sign is positive if a clockwise motion of atom 1 would superimpose it on atom 4 when the direction of view is from atom 2 to atom 3.

Supporting data

Least-squares mean planes (deviations in Å)

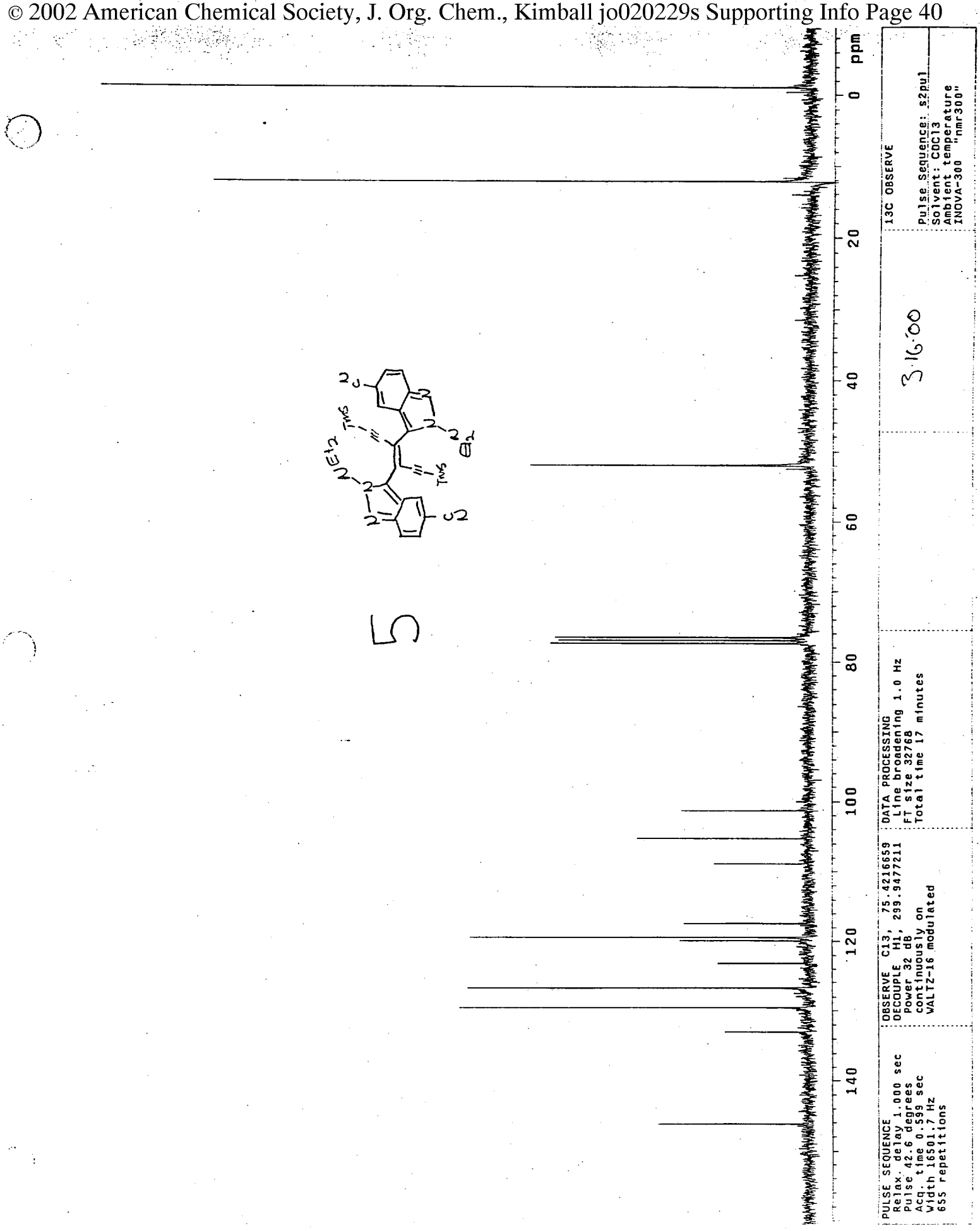
Plane 1	Defining atoms	Distance	Other atoms	Distance
	N(1)	-0.003(4)	N(3)	0.113
	N(2)	0.004(4)	N(4)	0.050
	C(1)	0.003(5)	C(9)	-0.159
	C(2)	0.011(5)	C(17)	0.013
	C(3)	0.014(5)		
	C(4)	0.006(5)		
	C(5)	-0.007(6)		
	C(6)	-0.022(5)		
	C(7)	0.010(5)		
	C(8)	-0.021(5)		

Mean deviation from plane: 0.010 Å; χ^2, 71.5

Plane 2	Defining atoms	Distance	Other atoms	Distance
	N(5)	-0.023(4)	N(7)	-0.041
	N(6)	-0.017(5)	N(8)	-0.137
	C(9)	0.038(5)	C(8)	0.290
	C(10)	-0.001(5)	C(18)	-0.075
	C(11)	0.014(5)		
	C(12)	-0.031(5)		
	C(13)	-0.026(5)		
	C(14)	0.029(6)		
	C(15)	0.030(6)		
	C(16)	0.002(5)		

Mean deviation from plane: 0.021 Å; χ^2, 245

Dihedral angle between least-squares planes: 6.0°



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