Crystal Structure of 5-(Dibenzylamino)-8-methoxy-4a-[1-(methoxyimino)ethyl]-2-phenyl-4a,5-dihydro[1,3,5]triazino[1,2-a]indole-1,3(2*H*,4*H*)-dione

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Naturally-occurring polyquinanes with fused five-membered carbocyclic frameworks have attracted attention due to their specific structures and biological activities.^{1,2} We have exploited the one-pot synthetic strategy for novel polycyclic hetero compounds such as hetero angular triquinane analogues³, hetero propellane⁴, indoles^{5,6}, indolines⁶, and imidazoquinazolinones⁷, using stereospecific skeletal rearrangement of *N*-phenyl-1,2-dihydrocinnoline-1,2-dicarboximide derivatives (urazoles) **1a-b**. In order to determine the stereo-chemistry of the hetero polyquinanes, X-ray analysis of the title compound **2** was undertaken.

The title compound **2** was synthesized by the photo-reaction of urazole **1b** with dibenzylamine in benzene (Fig. 1) according to the procedure reported previously.⁶ Colorless crystals (DMSO solvated) of **2** suitable for X-ray analysis were obtained by recrystallization from DMSO. An X-ray data collection was performed on a four-circle diffractometer with graphite-monochromated Cu K α radiation. The crystal data and the experimental details are shown in Table 1. The crystal structure was solved by the direct method with SIR92.⁸ The refined atomic parameters and equivalent isotropic temperature factors are given in Table 2. All hydrogen atoms were located based on the expected bonding geometry. All of the bond lengths and angles are reasonable. The selected ones are given in Tables 3 and 4. An ORTEP drawing of compound **2** is illustrated in Fig. 2.

The stereochemistry between dibenzylamino group and 1-((*E*)-methoxyimino)ethyl one was *trans*-configu-

ration. The torsion angle of N4-C2-C1-C13 is 139.8°. The dihedral angle between the least-squares planes of *cis*-fused triazine and indoline rings is 8.3°. The triazine ring becomes boat-form with the following derivations from the least-squares plane: C1, 0.3068 Å; N1, -0.1127 Å; C5, -0.0771 Å; N3, 0.1290 Å; C6, -0.0166 Å; N2, -0.1637 Å.

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Table 1 Crystal and experimental data

Formula: C ₃₆ H ₃₉ N ₅ O ₅ S
Formula weight=653.79
Crystal system: triclinic
Space group: <i>P</i> 1 <i>Z</i> =2
<i>a</i> =13.027(2) Å
<i>b</i> =13.516(4) Å
<i>c</i> =11.673(2) Å
α =106.64(2)
β =110.21(2)
γ =63.92(2)
<i>V</i> =1710.1(7) Å ³
<i>D</i> _{calc} =1.270 g/cm ³
μ (Cu K α)=12.42 cm ⁻¹
<i>T</i> =296 K
<i>F</i> (0 0 0)=692.00
Crystal dimensions(mm): 0.55×0.20×0.20
Radiation: graphite monochromated Cu K α
<i>R</i> =0.052
<i>R</i> _w =0.082
2 θ _{max} =120.2°
(Δ / σ) _{max} =0.07
($\Delta\rho$) _{max} =0.39 e Å ⁻³
($\Delta\rho$) _{min} =-0.43 e Å ⁻³
No. of reflections used=3662(<i>I</i> >3 σ (<i>I</i>))
No. of parameters=443
Measurement: Rigaku AFC7R
Program system: TEXSAN
Structure determination: direct method
Refinement: full matrix least-squares

Fig. 1 Reaction scheme.

Table 2 Atomic coordinates and equivalent isotropic displacement ($\text{\AA}^2$)

Atom	x	y	z	B_{eq}
O(1)	0.6055(2)	0.3371(2)	0.5437(2)	4.99(5)
O(2)	0.5742(2)	0.5308(2)	0.9263(2)	4.70(4)
O(3)	0.7998(2)	0.0920(2)	0.8631(2)	6.26(6)
O(4)	0.4532(2)	0.0441(2)	0.2541(2)	5.62(5)
N(1)	0.5149(2)	0.2955(2)	0.6476(2)	3.65(4)
N(2)	0.4869(2)	0.4045(2)	0.8404(2)	3.78(5)
N(3)	0.6097(2)	0.4189(2)	0.7457(2)	3.77(5)
N(4)	0.2825(2)	0.3580(2)	0.7447(2)	3.89(5)
N(5)	0.6999(2)	0.1649(2)	0.7900(2)	4.63(5)
C(1)	0.5015(2)	0.2945(2)	0.7669(2)	3.46(5)
C(2)	0.3915(2)	0.2621(2)	0.7294(2)	3.69(5)
C(3)	0.3954(2)	0.1997(2)	0.5994(2)	3.79(5)
C(4)	0.4668(2)	0.2224(2)	0.5548(2)	3.70(5)
C(5)	0.5785(2)	0.3474(2)	0.6369(2)	3.76(5)
C(6)	0.5574(2)	0.4549(2)	0.8438(2)	3.63(5)
C(7)	0.6964(2)	0.4598(2)	0.7501(2)	3.70(5)
C(8)	0.8088(3)	0.4165(2)	0.8247(3)	4.69(6)
C(9)	0.8942(3)	0.4512(3)	0.8279(4)	6.20(8)
C(10)	0.8682(3)	0.5277(3)	0.7543(4)	6.41(10)
C(11)	0.7576(3)	0.5704(3)	0.6815(4)	6.00(9)
C(12)	0.6698(3)	0.5368(2)	0.6785(3)	4.84(7)
C(13)	0.6124(2)	0.2079(2)	0.8367(2)	3.86(5)
C(14)	0.6107(3)	0.1885(3)	0.9550(3)	5.58(7)
C(15)	0.8976(3)	0.0596(4)	0.8124(5)	8.3(1)
C(16)	0.3421(2)	0.1263(2)	0.5246(3)	4.33(6)
C(17)	0.3609(2)	0.0783(2)	0.4084(3)	4.66(6)
C(18)	0.4352(2)	0.1009(2)	0.3678(2)	4.39(6)
C(19)	0.4900(2)	0.1747(2)	0.4399(2)	4.06(5)
C(20)	0.5384(3)	0.0526(3)	0.2145(3)	6.61(9)
C(21)	0.2392(2)	0.4381(2)	0.6599(3)	4.36(6)
C(22)	0.1489(2)	0.5487(2)	0.7019(3)	4.33(6)
C(23)	0.0521(3)	0.6065(3)	0.6167(3)	5.24(7)
C(24)	-0.0313(3)	0.7078(3)	0.6526(4)	6.04(8)
C(25)	-0.0183(4)	0.7522(3)	0.7747(4)	6.83(10)
C(26)	0.0780(4)	0.6979(3)	0.8606(4)	7.11(9)
C(27)	0.1615(3)	0.5956(3)	0.8253(3)	5.88(8)
C(28)	0.1869(2)	0.3226(2)	0.7334(3)	4.49(6)
C(29)	0.2101(2)	0.2589(2)	0.8316(3)	4.22(6)
C(30)	0.1765(3)	0.1688(3)	0.8006(3)	5.27(7)
C(31)	0.1894(3)	0.1132(3)	0.8920(4)	6.40(9)
C(32)	0.2373(4)	0.1454(4)	1.0110(4)	7.0(1)
C(33)	0.2729(4)	0.2331(3)	1.0437(3)	6.77(9)
C(34)	0.2594(3)	0.2905(3)	0.9548(3)	5.45(7)
S(1)	0.8278(3)	0.1287(2)	0.4073(2)	11.42(7)
S(1')	0.9001(8)	0.1414(9)	0.422(1)	10.1(3)
O(5)	0.9150(10)	0.0487(8)	0.3439(9)	17.1(3)
O(5')	0.827(3)	0.043(1)	0.414(2)	20.5(8)
C(35)	0.7477(7)	0.2337(8)	0.3256(5)	12.7(2)
C(36)	0.8913(5)	0.2046(6)	0.5395(6)	10.3(2)

$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} (a_i \cdot a_j).$$

Fig. 2 ORTEP drawing of the title compound with the atomic labeling scheme.

Table 3 Selected bond lengths ($\text{\AA}$)

Atom	Atom	Distance
O(1)	C(5)	1.208(3)
O(2)	C(6)	1.231(3)
N(1)	C(1)	1.466(3)
N(1)	C(4)	1.416(3)
N(1)	C(5)	1.352(3)
N(2)	C(1)	1.455(3)
N(2)	C(6)	1.348(3)
N(3)	C(5)	1.413(3)
N(3)	C(6)	1.394(3)
C(1)	C(2)	1.564(3)
C(2)	C(3)	1.516(4)
C(3)	C(4)	1.381(4)
C(3)	C(16)	1.385(4)
C(4)	C(19)	1.386(4)
C(16)	C(17)	1.381(4)
C(17)	C(18)	1.383(4)
C(18)	C(19)	1.395(4)

Table 4 Selected bond angles ($^\circ$)

Atom	Atom	Atom	Angle
C(1)	N(1)	C(4)	109.1(2)
C(1)	N(1)	C(5)	122.8(2)
C(4)	N(1)	C(5)	127.5(2)
C(1)	N(2)	C(6)	119.3(2)
C(5)	N(3)	C(6)	123.6(2)
C(5)	N(3)	C(7)	116.8(2)
C(6)	N(3)	C(7)	119.5(2)
C(2)	N(4)	C(21)	114.4(2)
N(1)	C(1)	N(2)	107.7(2)
N(1)	C(1)	C(2)	103.2(2)
N(2)	C(1)	C(2)	115.1(2)
N(4)	C(2)	C(1)	113.2(2)
N(4)	C(2)	C(3)	117.4(2)
C(1)	C(2)	C(3)	100.8(2)
C(2)	C(3)	C(4)	110.8(2)
C(2)	C(3)	C(16)	130.8(2)
C(4)	C(3)	C(16)	118.4(2)
N(1)	C(4)	C(3)	108.7(2)
N(1)	C(4)	C(19)	127.3(2)
C(3)	C(4)	C(19)	123.9(2)
O(1)	C(5)	N(1)	124.6(2)
O(1)	C(5)	N(3)	121.7(2)
N(1)	C(5)	N(3)	113.7(2)
O(2)	C(6)	N(2)	123.3(2)
O(2)	C(6)	N(3)	120.1(2)
N(2)	C(6)	N(3)	116.5(2)

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