Crystal Structure of 2,8,14-Trimethyl-2,8,14-triazatetracyclo-[14^{2,24},7,2^{10,13}]tetracosa-4,6,10,12,16,18,19,21,23-nonaene-3,9,15-trione

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Aromatic *N*-methylamides generally exist in a *cis*-amide form both as crystals and in solution.¹⁻³ A cyclic structure containing *N*-methylamide can be easily constructed, since the *cis*

preference arranges two moieties attached to the *N*-methylated amide close to each other. In this paper, we report on the crystal structure of 2,8,14-trimethyl-2,8,14-triazatetracyclo-[14^{2,24},7,2^{10,13}]tetracosa-4,6,10,12,16,18,19,21,23-nonaene-3,9,15-trione (**1**, Fig. 1), which was synthesized by a one-pot reaction (43% yield) from 4-(methylamino)benzoic acid under

Fig. 1 Chemical structure of the title compound.

Table 1 Crystal and experimental data

Formula: C ₂₄ H ₂₁ O ₃ N ₃
Formula weight = 399.45
Temperature: 296 K
Crystal size: 0.15 × 0.20 × 0.20 mm
Crystal color: colorless
Crystal description: prismatic
Crystal system: monoclinic
Space group: <i>P</i> 2 ₁ / <i>n</i> <i>Z</i> = 4
<i>a</i> = 7.8894(8) Å
<i>b</i> = 28.687(3) Å β = 94.200(7)°
<i>c</i> = 9.0405(5) Å
<i>V</i> = 2040.6(3) Å ³
<i>D_x</i> = 1.300 g/cm ³
No. of reflections collected = 3998
No. of reflections used = 3183
2θ _{max} = 135.2° with Cu Kα
<i>R</i> , <i>wR</i> = 0.052, 0.141
Goodness-of-fit = 2.23
(Δ/ <i>σ</i>) _{max} final cycle: 0.001
No. of parameters = 181

Table 2 Atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} /Å ²
O(1)	0.2845(2)	0.25064(6)	-0.2306(2)	5.04(4)
O(2)	0.0157(2)	0.13116(7)	0.5802(2)	5.39(4)
O(3)	0.8730(2)	0.02735(6)	0.1598(2)	6.18(5)
N(1)	0.7462(2)	0.07666(6)	-0.0071(2)	3.60(4)
N(2)	0.2239(2)	0.24783(6)	0.0108(2)	3.69(4)
N(3)	0.1988(2)	0.08174(6)	0.4835(2)	3.75(4)
C(1)	0.6329(3)	0.11548(6)	-0.0342(2)	3.16(4)
C(2)	0.5066(3)	0.11363(8)	-0.1486(2)	4.07(5)
C(3)	0.3991(3)	0.15114(8)	-0.1747(2)	4.05(5)
C(4)	0.4128(3)	0.18999(7)	-0.0834(2)	3.09(4)
C(5)	0.5402(2)	0.19157(6)	0.0301(2)	2.94(4)
C(6)	0.6522(2)	0.15502(7)	0.0526(2)	2.99(4)
C(7)	0.2988(3)	0.23138(7)	-0.1098(2)	3.46(4)
C(8)	0.1912(2)	0.21781(7)	0.1318(2)	3.24(4)
C(9)	0.2284(3)	0.23133(7)	0.2779(2)	3.86(5)
C(10)	0.2002(3)	0.20090(8)	0.3927(2)	3.81(5)
C(11)	0.1399(2)	0.15616(7)	0.3616(2)	3.25(4)
C(12)	0.0979(3)	0.14371(7)	0.2157(2)	3.43(4)
C(13)	0.1193(3)	0.17466(8)	0.1020(2)	3.51(4)
C(14)	0.1130(3)	0.12272(7)	0.4845(2)	3.47(4)
C(15)	0.3381(3)	0.07355(6)	0.3928(2)	3.05(4)
C(16)	0.3145(3)	0.04852(8)	0.2622(2)	3.95(5)
C(17)	0.4483(3)	0.04276(8)	0.1740(2)	4.17(5)
C(18)	0.6078(3)	0.05965(6)	0.2174(2)	3.10(4)
C(19)	0.6332(3)	0.08247(8)	0.3517(2)	3.79(5)
C(21)	0.7545(3)	0.05307(7)	0.1218(2)	3.62(4)
C(22)	0.1528(4)	0.29498(9)	0.0058(4)	5.20(6)
C(23)	0.1664(4)	0.04603(10)	0.5938(3)	5.00(6)
C(24)	0.8777(4)	0.06916(10)	-0.1110(3)	4.87(6)

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Fig. 2 ORTEP drawing of the title compound with atom labeling.

the same condition (reflux with 2.0 eq. of tetrachlorosilane in pyridine for 48 h) as we previously reported for the cyclization of 3-(methylamino)benzoic acid.⁴

Prismatic single crystals of compound **1** were obtained by recrystallization from ethyl acetate. Intensity data were collected on a Rigaku AFC5R diffractometer using Cu K α radiation monochromated with graphite at 23°C. The crystal data are summarized in Table 1. The structure was solved by a direct method (SHELX97),⁵ expanded by DIRDIF94 and refined by a full-matrix least-squares method with anisotropic thermal parameters for the non-hydrogen atoms. All hydrogen atoms indicated in the ORTEP drawing in Fig. 2 were fixed at geometrically reasonable positions. The bond length and selected bond angles are listed in Table 3. Each molecule in the unit cell has a small cavity surrounded by three benzene rings and no symmetry element in itself. One of the methyl groups (C22) directs to the cavity of the other molecule. All of the amide bonds are nearly planar; the torsion angles of C1-N1-C21-C18, C8-N2-C7-C4, C15-N3-C14-C11 are 17.2°, -27.1°, 14.1°, respectively, and the three phenylene rings are tilted from orthogonal planes to the amide planes; the torsion angles of N1-C21-C18-C17, N2-C7-C4-C5, N3-C14-C11-C12 are 68.3°, -51.9°, 60.8°, respectively.

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Table 3 Selected bond distances (Å) and angles (°)

Atom	Atom	Distance	Atom	Atom	Distance
O(1)	C(7)	1.222(2)	O(2)	C(14)	1.222(2)
O(3)	C(21)	1.220(3)	N(1)	C(1)	1.438(2)
N(1)	C(21)	1.345(3)	C(11)	C(14)	1.495(3)
N(1)	C(24)	1.466(3)	N(2)	C(7)	1.362(3)
N(2)	C(8)	1.430(3)	N(2)	C(22)	1.464(3)
N(3)	C(14)	1.357(3)	N(3)	C(15)	1.439(2)
N(3)	C(23)	1.464(3)	C(18)	C(21)	1.506(3)
C(4)	C(7)	1.499(3)			

Atom	Atom	Atom	Angle	Atom	Atom	Atom	Angle
C(1)	N(1)	C(21)	121.8(1)	C(1)	N(1)	C(24)	117.7(2)
C(21)	N(1)	C(24)	119.3(2)	C(7)	N(2)	C(8)	121.1(2)
C(7)	N(2)	C(22)	118.9(2)	C(10)	C(11)	C(14)	120.5(2)
C(8)	N(2)	C(22)	119.3(2)	C(12)	C(11)	C(14)	120.4(2)
C(14)	N(3)	C(15)	123.2(2)	C(14)	N(3)	C(23)	119.3(2)
C(15)	N(3)	C(23)	116.9(2)	O(2)	C(14)	N(3)	121.1(2)
N(1)	C(1)	C(2)	120.1(2)	O(2)	C(14)	C(11)	122.0(2)
N(1)	C(1)	C(6)	119.9(2)	N(3)	C(14)	C(11)	116.9(2)
N(3)	C(15)	C(16)	120.7(2)	N(3)	C(15)	C(20)	119.7(2)
C(3)	C(4)	C(7)	121.5(2)	C(5)	C(4)	C(7)	119.2(2)
C(17)	C(18)	C(21)	121.1(2)	C(19)	C(18)	C(21)	119.7(2)
O(1)	C(7)	N(2)	122.8(2)	O(1)	C(7)	C(4)	121.1(2)
N(2)	C(7)	C(4)	116.0(2)	O(3)	C(21)	N(1)	122.4(2)
N(2)	C(8)	C(9)	121.5(2)	O(3)	C(21)	C(18)	121.3(2)
N(2)	C(8)	C(13)	119.0(2)	N(1)	C(21)	C(18)	116.3(2)

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