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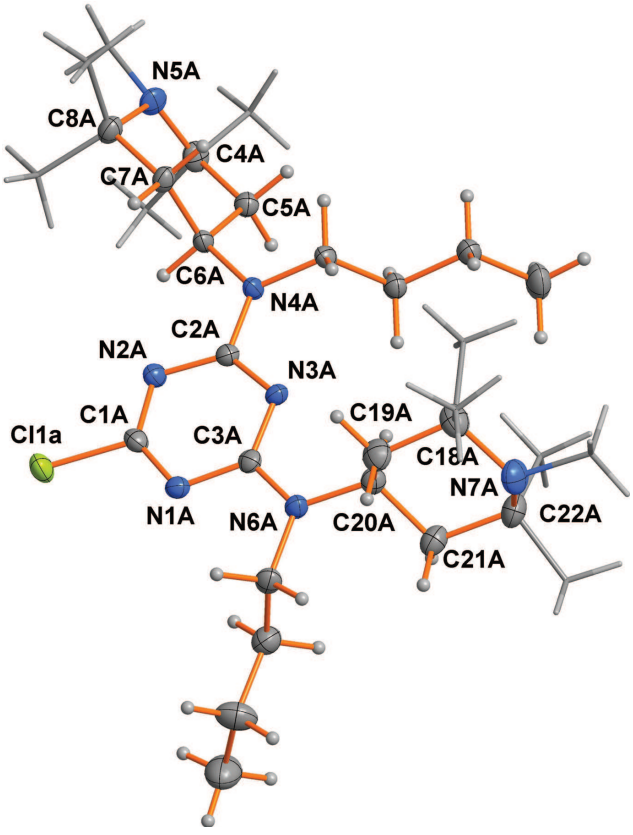
Crystal structure of N^2,N^4-dibutyl-6-chloro-N^2,N^4-bis(1,2,2,6,6-pentamethylpiperidin-4-yl)-1,3,5-triazine-2,4-diamine, $C_{31}H_{58}ClN_7$ – Important intermediate of Chimassorb 119 synthesis



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Abstract

$C_{31}H_{58}ClN_7$, monoclinic, $P2_1/c$ (no. 14), $a = 29.9370(18)$ Å, $b = 15.6432(8)$ Å, $c = 14.2792(8)$ Å, $\beta = 90.819(2)°$, $V = 6686.4(6)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0671$, $wR_{ref}(F^2) = 0.1925$, $T = 173(2)$ K.

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One of two crystallographically independent molecules of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.43 × 0.31 × 0.27 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.15 mm^{-1}
Diffractometer, scan mode:	PHOTON100 CMOS, ω
θ_{max}, completeness:	25.4°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	32658, 12056, 0.072
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 7336
$N(param)_{refined}$:	727
Programs:	Bruker [3], SHELX [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1A	0.26829(11)	0.3979(2)	0.1596(2)	0.0262(8)
C2A	0.33707(11)	0.4313(2)	0.1164(2)	0.0235(7)
C3A	0.31006(11)	0.4797(2)	0.2537(2)	0.0238(7)
C4A	0.40603(13)	0.2744(2)	−0.1439(3)	0.0323(9)
C5A	0.41011(11)	0.3296(2)	−0.0560(2)	0.0271(8)
H5A1	0.4197	0.2933	−0.0027	0.032*
H5A2	0.4335	0.3733	−0.0657	0.032*
C6A	0.36684(11)	0.3740(2)	−0.0315(2)	0.0244(8)
H6A	0.3437	0.3292	−0.0204	0.029*
C7A	0.35167(12)	0.4278(2)	−0.1147(2)	0.0273(8)
H7A1	0.3739	0.4735	−0.1251	0.033*
H7A2	0.3229	0.4555	−0.0996	0.033*
C8A	0.34571(12)	0.3762(2)	−0.2055(2)	0.0327(9)
C9A	0.38706(17)	0.2823(3)	−0.3125(3)	0.0560(12)
H9A1	0.4169	0.2594	−0.3247	0.084*
H9A2	0.3786	0.3227	−0.3621	0.084*
H9A3	0.3654	0.2353	−0.3112	0.084*
C10A	0.37989(16)	0.1909(2)	−0.1225(3)	0.0505(11)
H10A	0.3512	0.2054	−0.0941	0.076*
H10B	0.3974	0.1555	−0.0789	0.076*
H10C	0.3745	0.1593	−0.1808	0.076*
C11A	0.45398(14)	0.2478(3)	−0.1692(3)	0.0508(12)
H11A	0.4528	0.2026	−0.2168	0.076*
H11B	0.4696	0.2265	−0.1131	0.076*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H11C	0.4700	0.2973	−0.1939	0.076*
C12A	0.30264(13)	0.3226(3)	−0.2003(3)	0.0467(11)
H12A	0.3020	0.2809	−0.2515	0.070*
H12B	0.2766	0.3603	−0.2059	0.070*
H12C	0.3019	0.2924	−0.1402	0.070*
C13A	0.33966(16)	0.4421(3)	−0.2842(3)	0.0507(11)
H13A	0.3682	0.4706	−0.2956	0.076*
H13B	0.3174	0.4846	−0.2659	0.076*
H13C	0.3295	0.4131	−0.3416	0.076*
C14A	0.40926(11)	0.4853(2)	0.0654(2)	0.0249(8)
H14A	0.3983	0.5406	0.0897	0.030*
H14B	0.4220	0.4960	0.0029	0.030*
C15A	0.44614(11)	0.4529(2)	0.1302(2)	0.0291(8)
H15A	0.4574	0.3978	0.1059	0.035*
H15B	0.4336	0.4422	0.1929	0.035*
C16A	0.48495(12)	0.5155(2)	0.1396(3)	0.0329(9)
H16A	0.4978	0.5254	0.0770	0.040*
H16B	0.4735	0.5709	0.1627	0.040*
C17A	0.52144(13)	0.4847(3)	0.2053(3)	0.0478(11)
H17A	0.5091	0.4751	0.2675	0.072*
H17B	0.5451	0.5279	0.2094	0.072*
H17C	0.5339	0.4311	0.1815	0.072*
C18A	0.38858(13)	0.7230(2)	0.3488(3)	0.0403(10)
C19A	0.34609(13)	0.6689(2)	0.3393(3)	0.0402(10)
H19A	0.3242	0.6884	0.3859	0.048*
H19B	0.3328	0.6777	0.2761	0.048*
C20A	0.35516(12)	0.5747(2)	0.3537(2)	0.0284(8)
H20A	0.3784	0.5572	0.3078	0.034*
C21A	0.37470(13)	0.5622(2)	0.4509(3)	0.0361(9)
H21A	0.3806	0.5006	0.4612	0.043*
H21B	0.3526	0.5809	0.4976	0.043*
C22A	0.41811(14)	0.6121(3)	0.4662(3)	0.0402(10)
C23A	0.44883(18)	0.7587(3)	0.4616(4)	0.0685(14)
H23A	0.4599	0.7471	0.5252	0.103*
H23B	0.4398	0.8188	0.4566	0.103*
H23C	0.4725	0.7469	0.4166	0.103*
C24A	0.41844(17)	0.7103(3)	0.2629(3)	0.0614(13)
H24A	0.4463	0.7422	0.2718	0.092*
H24B	0.4028	0.7312	0.2066	0.092*
H24C	0.4252	0.6493	0.2555	0.092*
C25A	0.37391(17)	0.8176(3)	0.3495(4)	0.0669(15)
H25A	0.3599	0.8308	0.4094	0.100*
H25B	0.3525	0.8278	0.2982	0.100*
H25C	0.4001	0.8544	0.3414	0.100*
C26A	0.45685(14)	0.5689(3)	0.4134(4)	0.0600(13)
H26A	0.4485	0.5624	0.3471	0.090*
H26B	0.4629	0.5126	0.4407	0.090*
H26C	0.4837	0.6045	0.4188	0.090*
C27A	0.42895(19)	0.6066(3)	0.5716(3)	0.0699(15)
H27A	0.4592	0.6282	0.5836	0.105*
H27B	0.4270	0.5470	0.5921	0.105*
H27C	0.4075	0.6412	0.6063	0.105*
C28A	0.27865(12)	0.5212(2)	0.4033(2)	0.0311(8)
H28A	0.2498	0.5297	0.3698	0.037*
H28B	0.2828	0.5695	0.4475	0.037*
C29A	0.27680(13)	0.4387(2)	0.4581(3)	0.0371(9)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H29A	0.2735	0.3905	0.4136	0.044*
H29B	0.3054	0.4310	0.4926	0.044*
C30A	0.23909(16)	0.4356(3)	0.5267(3)	0.0532(12)
H30A	0.2448	0.4786	0.5764	0.064*
H30B	0.2110	0.4519	0.4937	0.064*
C31A	0.23274(18)	0.3507(3)	0.5714(3)	0.0629(13)
H31A	0.2238	0.3089	0.5236	0.094*
H31B	0.2094	0.3548	0.6187	0.094*
H31C	0.2608	0.3324	0.6013	0.094*
C1B	0.22620(11)	0.8738(2)	0.1645(2)	0.0244(8)
C2B	0.15926(11)	0.9183(2)	0.1164(2)	0.0208(7)
C3B	0.18819(11)	0.9627(2)	0.2562(2)	0.0216(7)
C4B	0.14532(12)	0.8773(2)	−0.2102(2)	0.0271(8)
C5B	0.14263(11)	0.9247(2)	−0.1166(2)	0.0235(7)
H5B1	0.1724	0.9490	−0.1013	0.028*
H5B2	0.1214	0.9729	−0.1237	0.028*
C6B	0.12786(11)	0.8691(2)	−0.0352(2)	0.0232(7)
H6B	0.1504	0.8226	−0.0261	0.028*
C7B	0.08329(11)	0.8281(2)	−0.0607(2)	0.0250(8)
H7B1	0.0603	0.8734	−0.0658	0.030*
H7B2	0.0746	0.7892	−0.0095	0.030*
C8B	0.08388(12)	0.7776(2)	−0.1528(2)	0.0278(8)
C9B	0.09836(15)	0.7941(3)	−0.3216(2)	0.0456(11)
H9B1	0.1194	0.7464	−0.3254	0.068*
H9B2	0.1056	0.8368	−0.3693	0.068*
H9B3	0.0679	0.7731	−0.3324	0.068*
C10B	0.18703(13)	0.8199(2)	−0.2117(3)	0.0386(9)
H10D	0.1886	0.7859	−0.1540	0.058*
H10E	0.2138	0.8556	−0.2160	0.058*
H10F	0.1853	0.7816	−0.2659	0.058*
C11B	0.15117(14)	0.9473(2)	−0.2845(2)	0.0404(10)
H11D	0.1582	0.9208	−0.3447	0.061*
H11E	0.1756	0.9855	−0.2654	0.061*
H11F	0.1234	0.9802	−0.2909	0.061*
C12B	0.10848(14)	0.6921(2)	−0.1379(3)	0.0396(10)
H12D	0.1107	0.6620	−0.1978	0.059*
H12E	0.0919	0.6568	−0.0936	0.059*
H12F	0.1385	0.7031	−0.1126	0.059*
C13B	0.03484(13)	0.7566(2)	−0.1777(3)	0.0388(10)
H13D	0.0200	0.8079	−0.2022	0.058*
H13E	0.0195	0.7370	−0.1214	0.058*
H13F	0.0338	0.7114	−0.2253	0.058*
C14B	0.08858(11)	0.9799(2)	0.0647(2)	0.0252(8)
H14C	0.0753	0.9935	0.0025	0.030*
H14D	0.1007	1.0337	0.0915	0.030*
C15B	0.05205(11)	0.9468(2)	0.1279(2)	0.0274(8)
H15C	0.0392	0.8940	0.1003	0.033*
H15D	0.0653	0.9319	0.1896	0.033*
C16B	0.01503(11)	1.0112(2)	0.1416(2)	0.0297(8)
H16C	0.0021	1.0266	0.0796	0.036*
H16D	0.0279	1.0638	0.1695	0.036*
C17B	−0.02210(13)	0.9785(3)	0.2041(3)	0.0413(10)
H17D	−0.0366	0.9291	0.1744	0.062*
H17E	−0.0442	1.0239	0.2133	0.062*
H17F	−0.0094	0.9615	0.2649	0.062*
C18B	0.08348(12)	1.1103(2)	0.4588(2)	0.0308(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C19B	0.12403(12)	1.0513(2)	0.4462(2)	0.0291(8)
H19C	0.1458	1.0616	0.4979	0.035*
H19D	0.1141	0.9910	0.4501	0.035*
C20B	0.14706(11)	1.0654(2)	0.3529(2)	0.0273(8)
H20B	0.1244	1.0560	0.3018	0.033*
C21B	0.16195(13)	1.1572(2)	0.3482(3)	0.0342(9)
H21C	0.1772	1.1667	0.2880	0.041*
H21D	0.1839	1.1681	0.3993	0.041*
C22B	0.12328(14)	1.2214(2)	0.3563(3)	0.0370(9)
C23B	0.06216(17)	1.2621(3)	0.4622(4)	0.0603(14)
H23D	0.0375	1.2546	0.4171	0.090*
H23E	0.0741	1.3202	0.4569	0.090*
H23F	0.0512	1.2531	0.5258	0.090*
C24B	0.04405(13)	1.0789(3)	0.3974(3)	0.0450(10)
H24D	0.0198	1.1210	0.3991	0.068*
H24E	0.0333	1.0240	0.4213	0.068*
H24F	0.0539	1.0718	0.3328	0.068*
C25B	0.07045(15)	1.1009(3)	0.5620(3)	0.0476(11)
H25D	0.0934	1.1273	0.6020	0.071*
H25E	0.0679	1.0401	0.5776	0.071*
H25F	0.0417	1.1292	0.5721	0.071*
C26B	0.09587(16)	1.2217(3)	0.2646(3)	0.0515(12)
H26D	0.0855	1.1636	0.2507	0.077*
H26E	0.1146	1.2421	0.2134	0.077*
H26F	0.0700	1.2596	0.2711	0.077*
C27B	0.14392(17)	1.3105(3)	0.3695(4)	0.0569(13)
H27D	0.1208	1.3541	0.3606	0.085*
H27E	0.1675	1.3190	0.3236	0.085*
H27F	0.1567	1.3152	0.4329	0.085*
C28B	0.21869(11)	0.9933(2)	0.4116(2)	0.0293(8)
H28C	0.2181	1.0426	0.4551	0.035*
H28D	0.2486	0.9915	0.3829	0.035*
C29B	0.21142(13)	0.9108(2)	0.4672(2)	0.0363(9)
H29C	0.1807	0.9114	0.4921	0.044*
H29D	0.2135	0.8617	0.4238	0.044*
C30B	0.24411(14)	0.8964(3)	0.5486(3)	0.0433(10)
H30C	0.2446	0.9484	0.5882	0.052*
H30D	0.2329	0.8488	0.5874	0.052*
C31B	0.29019(15)	0.8767(3)	0.5207(4)	0.0565(12)
H31D	0.2900	0.8275	0.4782	0.085*
H31E	0.3083	0.8632	0.5765	0.085*
H31F	0.3030	0.9263	0.4888	0.085*
Cl1A	0.21671(3)	0.35110(6)	0.13449(7)	0.0368(2)
Cl1B	0.27567(3)	0.81828(6)	0.14199(7)	0.0403(3)
N1A	0.26999(9)	0.43953(18)	0.23970(18)	0.0260(7)
N2A	0.29914(9)	0.38719(17)	0.09507(19)	0.0259(7)
N3A	0.34409(9)	0.47881(17)	0.19321(18)	0.0238(6)
N4A	0.37118(9)	0.42679(18)	0.05467(18)	0.0245(6)
N5A	0.38746(10)	0.32660(19)	−0.2215(2)	0.0332(7)
N6A	0.31499(9)	0.52225(18)	0.33512(18)	0.0267(7)
N7A	0.41027(11)	0.7038(2)	0.4410(2)	0.0414(8)
N1B	0.22630(9)	0.91616(18)	0.24427(19)	0.0266(7)
N2B	0.19552(9)	0.86809(17)	0.09815(19)	0.0256(6)
N3B	0.15456(9)	0.96737(17)	0.19276(18)	0.0230(6)
N4B	0.12569(9)	0.91909(17)	0.05227(18)	0.0225(6)
N5B	0.10165(10)	0.83328(18)	−0.22820(19)	0.0277(7)
N6B	0.18456(9)	1.00567(18)	0.33765(19)	0.0273(7)
N7B	0.09753(11)	1.20000(19)	0.4427(2)	0.0359(8)

Source of material

The compound we report here can be easily synthesized by a known procedure mentioned in a patent [1]. Evaporation at room temperature yielded colourless crystals of the title compound.

Experimental details

The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 23, 0, 13 or 137 option). All H atoms were positioned geometrically and refined using a riding model: C—H = 0.93 and 0.96 Å for aromatic and CH_3 H atoms, respectively, with $U_{iso}(H) = k \times U_{eq}(N,C)$, where k = 1.5 for CH_3 H atoms and = 1.2 for other H atoms.

Comment

The title compound, we report here is an important intermediate of Chimassorb 119. Ciba Specialty Chemicals was the first company to improve the performance of the product by improving the average relative molecular quality of the products. Chimassorb 119 is one of them. It has been widely used as light stabilizer in polyolefins because of its high molecular weight.

The compound crystallizes in the non-centrosymmetric space group $P2_1/c$ (no. 14) with two crystallographically independent molecules. The molecular structure of the title compound is shown in Figure 1. In the title crystal structure,the bond lengths and angles are within normal ranges [2]. There are 6 rings, triazine rings *Cg*1 A (C1A—N3A—C3A—N1A—N2A—C2A) and *Cg*1B(C1B—N3B—C3B—N1B—N2B—C2B), piperidine rings *Cg*2 A (C8A—N5A—C7A—C6A—C5A—C4A) and *Cg*2B (C8B—N5B—C7B—C6B—C5B—C4B), piperidine rings *Cg*3 A (N7A—C18A—C19A—C20A—C21A—C22A) and *Cg*3B (N7B—C18B—C19B—C20B—C21B—C22B), they are not planar. The dihedral angle between the best planes of ring *Cg*1 A and ring *Cg*2 A is 66.6°. In the second molecule of the crystal, the corresponding dihedral angle is 70.8°. The rings *Cg*1 A and ring *Cg*3 A are nearly perpendicular to each other with a dihedral angle of 79.9°. In the second molecule of the crystal, the corresponding dihedral angle is 85.3°. The planes of the ring *Cg*2 A and ring *Cg*3 A show a dihedral angle of 110.6°, and the other one is 113.1°. Dimeric aggregates are formed through C30A—H30A$\cdots$*Cg*1B and C30B—H30C$\cdots$*Cg*1 A non-classical hydrogen bonds. The distances between the C30—H30 and the triazine ring centroid of *Cg*1(x, $3/2-y$, $1/2+z$) are 3.475(5) and 3.664(5), respectively. In the crystal structure, π−π stacking interactions are not found.

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