Crystal structure of dihydroergocristine bis(dioxane) solvate, $(C_{35}H_{41}N_5O_5)(C_4H_8O_2)_2$

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Source of material: Dihydroergocristine (Galena Co., Czech Republic) was dissolved in dioxane at 353 K (140 mg/2ml) and the solution was allowed to cool. After 24 hours crystals were separated, washed with dioxane and dried in air.

The title compound crystallizes as a solvate with two slightly disordered dioxane molecules.

$C_{43}H_{57}N_5O_9$, monoclinic, $P12_11$ (No. 4), $a = 10.0503(9)$ Å, $b = 10.553(1)$ Å, $c = 19.743(2)$ Å, $\beta = 92.954(7)^\circ$, $V = 2091.2$ Å³, $Z = 2$, $R(F) = 0.073$, $R_w(F) = 0.087$.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prisms, size 0.43 x 0.56 x 0.69 mm
Wavelength:	Cu $K\alpha$ radiation (1.54183 Å)
μ :	0.68 cm ⁻¹
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	2 θ
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{unique}}$:	2793
Criterion for F_o :	$F_o > 1.96 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	513
Program:	CRYSTALS

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(11)	2a	1.2385(6)	0.8365(8)	0.4929(3)	0.08446
H(31)	2a	1.1618(4)	1.0892(5)	0.4408(2)	0.06188
H(32)	2a	1.2019(4)	1.0421(5)	0.3658(2)	0.06188
H(41)	2a	0.9393(4)	1.1179(4)	0.4029(2)	0.04779
H(51)	2a	0.9406(4)	1.3874(4)	0.3011(2)	0.05363
H(52)	2a	0.8542(4)	1.3071(4)	0.3542(2)	0.05363
H(61)	2a	0.9044(3)	1.2282(4)	0.2184(2)	0.04538
H(71)	2a	0.7451(4)	1.0976(4)	0.3144(2)	0.05185
H(72)	2a	0.7766(4)	1.0447(4)	0.2391(2)	0.05185
H(81)	2a	1.0039(3)	1.0162(4)	0.2746(2)	0.04378
H(101)	2a	0.7453(5)	0.8409(5)	0.2711(2)	0.05891
H(111)	2a	0.7210(5)	0.6346(5)	0.3164(3)	0.06860
H(121)	2a	0.8725(5)	0.5531(5)	0.4035(3)	0.06962
H(151)	2a	1.1301(5)	1.3964(6)	0.3669(3)	0.07381
H(152)	2a	1.1992(5)	1.2707(6)	0.4019(3)	0.07381
H(153)	2a	1.0617(5)	1.3273(6)	0.4305(3)	0.07381
H(191)	2a	0.2575(4)	1.3368(4)	0.1118(2)	0.05098
H(211)	2a	0.0511(5)	1.6979(5)	0.1914(3)	0.07379
H(212)	2a	0.0601(5)	1.6355(5)	0.2671(3)	0.07379
H(221)	2a	0.1721(5)	1.8135(6)	0.2954(3)	0.07863
H(222)	2a	0.2113(5)	1.8397(6)	0.2174(3)	0.07863
H(231)	2a	0.4164(5)	1.7726(5)	0.2656(3)	0.07140
H(232)	2a	0.3453(5)	1.6672(5)	0.3124(3)	0.07140
H(241)	2a	0.3739(4)	1.6566(4)	0.1689(2)	0.04608
H(261)	2a	0.7197(4)	1.4825(4)	0.0665(2)	0.04813
H(271)	2a	0.6019(5)	1.6593(5)	0.0215(2)	0.06444
H(272)	2a	0.5145(5)	1.6645(5)	0.0889(2)	0.06444
H(273)	2a	0.5016(5)	1.5428(5)	0.0380(2)	0.06444
H(281)	2a	0.8111(5)	1.6867(6)	0.0865(3)	0.08349
H(282)	2a	0.7364(5)	1.6930(6)	0.1579(3)	0.08349
H(283)	2a	0.8542(5)	1.5891(6)	0.1479(3)	0.08349
H(291)	2a	0.3019(6)	1.2513(5)	0.2472(3)	0.08023
H(292)	2a	0.1511(6)	1.2302(5)	0.2144(3)	0.08023
H(311)	2a	0.1200(9)	1.1180(7)	0.1096(4)	0.10007
H(321)	2a	0.170(1)	0.937(1)	0.0438(5)	0.16871
H(341)	2a	0.5341(9)	0.9043(7)	0.1546(7)	0.12592
H(351)	2a	0.4741(7)	1.0926(7)	0.2249(7)	0.11900
H(521)	2a	0.8796(8)	1.1958(9)	-0.0638(4)	0.10996
H(522)	2a	0.9676(8)	1.0917(9)	-0.0198(4)	0.10996
H(531)	2a	0.935(1)	1.2699(8)	0.0457(4)	0.11696
H(532)	2a	0.775(1)	1.2451(8)	0.0372(4)	0.11696
H(551)	2a	0.6786(8)	1.088(1)	0.0778(4)	0.13244
H(552)	2a	0.7595(8)	0.977(1)	0.1203(4)	0.13244
H(561)	2a	0.858(1)	0.916(1)	0.0236(4)	0.13423
H(562)	2a	0.696(1)	0.907(1)	0.0129(4)	0.13423
H(621)	2a	0.368(1)	0.605(2)	0.4116(5)	0.18075
H(622)	2a	0.416(1)	0.467(2)	0.4410(5)	0.18075
H(631)	2a	0.499(1)	0.634(3)	0.516(2)	0.28798
H(632)	2a	0.590(1)	0.615(3)	0.450(2)	0.28798
H(651)	2a	0.477(1)	0.523(2)	0.5952(5)	0.17658
H(652)	2a	0.506(1)	0.371(2)	0.5991(5)	0.17658

Table 2. (Continued)

Atom	Site	x	y	z	U_{iso}
H(661)	2a	0.283(1)	0.429(2)	0.5786(6)	0.18235
H(662)	2a	0.344(1)	0.358(2)	0.5135(6)	0.18235
H(331)	2a	0.376(1)	0.837(1)	0.0661(5)	0.16036
H(851)	2a	0.4875(3)	1.3867(3)	0.2851(1)	0.05000
H(731)	2a	0.8195(3)	1.3959(3)	0.1715(2)	0.05000
H(711)	2a	1.1056(5)	0.6323(5)	0.4848(2)	0.08272

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2a	1.0852(5)	0.7101(5)	0.4573(2)	0.100(3)	0.072(3)	0.078(3)	0.027(3)	0.001(2)	0.028(3)
N(2)	2a	1.0357(3)	1.2331(4)	0.3394(2)	0.042(2)	0.053(2)	0.061(2)	0.003(2)	-0.007(1)	0.006(2)
N(3)	2a	0.7314(3)	1.3840(3)	0.1850(2)	0.037(2)	0.047(2)	0.058(2)	0.016(1)	0.005(1)	0.011(2)
N(4)	2a	0.4003(3)	1.4090(3)	0.1681(1)	0.038(2)	0.044(2)	0.046(2)	0.006(1)	0.003(1)	0.005(1)
N(5)	2a	0.2048(3)	1.5693(4)	0.2045(2)	0.040(2)	0.047(2)	0.088(2)	0.013(2)	0.016(2)	0.004(2)
O(1)	2a	0.6147(3)	1.2790(3)	0.2623(2)	0.043(1)	0.061(2)	0.085(2)	0.015(1)	0.011(1)	0.023(2)
O(2)	2a	0.5678(2)	1.5397(3)	0.2061(1)	0.043(1)	0.051(2)	0.050(1)	0.008(1)	0.001(1)	-0.003(1)
O(3)	2a	0.5043(3)	1.3020(3)	0.0845(1)	0.067(2)	0.053(2)	0.061(2)	0.002(1)	0.013(1)	-0.012(2)
O(4)	2a	0.0432(3)	1.4320(4)	0.1719(2)	0.041(2)	0.066(2)	0.157(3)	-0.002(2)	0.019(2)	0.007(3)
O(5)	2a	0.4304(3)	1.4561(3)	0.2852(1)	0.069(2)	0.062(2)	0.042(1)	0.021(2)	0.011(1)	0.010(1)
C(1)	2a	1.1575(6)	0.8211(8)	0.4625(3)	0.085(4)	0.120(6)	0.065(3)	0.039(4)	-0.012(3)	0.024(3)
C(2)	2a	1.0988(4)	0.9085(6)	0.4176(2)	0.056(3)	0.076(3)	0.059(2)	0.022(3)	-0.004(2)	0.015(3)
C(3)	2a	1.1303(4)	1.0419(5)	0.3991(2)	0.053(2)	0.066(3)	0.061(2)	0.002(2)	-0.014(2)	0.008(2)
C(4)	2a	1.0050(4)	1.1062(4)	0.3670(2)	0.049(2)	0.047(2)	0.045(2)	0.005(2)	-0.003(2)	0.002(2)
C(5)	2a	0.9158(4)	1.3005(4)	0.3161(2)	0.044(2)	0.047(2)	0.065(2)	0.005(2)	-0.001(2)	-0.006(2)
C(6)	2a	0.8452(3)	1.2291(4)	0.2576(2)	0.043(2)	0.044(2)	0.052(2)	0.011(2)	0.001(2)	0.004(2)
C(7)	2a	0.8132(4)	1.0936(4)	0.2793(2)	0.044(2)	0.049(2)	0.056(2)	0.008(2)	-0.007(2)	-0.001(2)
C(8)	2a	0.9367(3)	1.0254(4)	0.3099(2)	0.043(2)	0.047(2)	0.049(2)	0.010(2)	0.000(2)	0.006(2)
C(9)	2a	0.9051(4)	0.8937(4)	0.3331(2)	0.050(2)	0.050(2)	0.047(2)	0.013(2)	0.002(2)	-0.001(2)
C(10)	2a	0.8084(5)	0.8119(5)	0.3087(2)	0.066(3)	0.055(3)	0.068(3)	0.013(2)	-0.005(2)	-0.001(2)
C(11)	2a	0.7961(5)	0.6891(5)	0.3344(3)	0.075(3)	0.046(3)	0.089(3)	-0.004(2)	0.010(3)	-0.009(3)
C(12)	2a	0.8808(5)	0.6414(5)	0.3858(3)	0.095(4)	0.046(2)	0.079(3)	0.006(3)	0.021(3)	0.005(3)
C(13)	2a	0.9806(5)	0.7206(5)	0.4099(2)	0.081(3)	0.062(3)	0.057(2)	0.028(3)	0.014(2)	0.018(2)
C(14)	2a	0.9910(4)	0.8465(4)	0.3849(2)	0.052(2)	0.050(2)	0.061(2)	0.013(2)	0.008(2)	0.006(2)
C(15)	2a	1.1126(5)	1.3131(6)	0.3890(3)	0.068(3)	0.067(3)	0.098(4)	-0.015(3)	-0.021(3)	-0.005(3)
C(16)	2a	0.7197(3)	1.2999(4)	0.2355(2)	0.036(2)	0.045(2)	0.058(2)	0.009(2)	0.001(2)	0.010(2)
C(17)	2a	0.6231(3)	1.4592(4)	0.1555(2)	0.040(2)	0.042(2)	0.045(2)	0.012(2)	0.009(1)	0.001(2)
C(18)	2a	0.5029(4)	1.3760(4)	0.1308(2)	0.048(2)	0.038(2)	0.045(2)	0.015(2)	0.009(2)	0.007(2)
C(19)	2a	0.2653(4)	1.3603(4)	0.1610(2)	0.049(2)	0.037(2)	0.074(3)	0.004(2)	0.014(2)	0.009(2)
C(20)	2a	0.1622(4)	1.4594(5)	0.1795(2)	0.044(2)	0.052(3)	0.089(3)	0.007(2)	0.019(2)	0.015(2)
C(21)	2a	0.1130(5)	1.6680(5)	0.2293(3)	0.061(3)	0.064(3)	0.113(4)	0.020(3)	0.039(3)	0.001(3)
C(22)	2a	0.2043(5)	1.7736(6)	0.2534(3)	0.064(3)	0.067(3)	0.095(3)	0.020(3)	0.021(2)	-0.008(3)
C(23)	2a	0.3414(5)	1.7105(5)	0.2673(3)	0.065(3)	0.065(3)	0.074(3)	0.021(2)	0.008(2)	-0.021(3)
C(24)	2a	0.3419(4)	1.6147(4)	0.2105(2)	0.047(2)	0.043(2)	0.058(2)	0.017(2)	0.010(2)	-0.004(2)
C(25)	2a	0.4365(3)	1.5019(4)	0.2201(2)	0.038(2)	0.051(2)	0.042(2)	0.012(2)	0.001(1)	-0.002(2)
C(26)	2a	0.6745(4)	1.5400(4)	0.0983(2)	0.043(2)	0.052(2)	0.054(2)	0.006(2)	0.008(2)	0.005(2)
C(27)	2a	0.5639(5)	1.6078(5)	0.0584(2)	0.063(3)	0.068(3)	0.065(3)	0.013(2)	0.003(2)	0.027(2)
C(28)	2a	0.7780(5)	1.6355(6)	0.1248(3)	0.067(3)	0.075(4)	0.109(4)	-0.015(3)	0.001(3)	0.027(3)
C(29)	2a	0.2471(6)	1.2381(5)	0.2041(3)	0.088(3)	0.043(3)	0.103(4)	0.003(3)	0.040(3)	0.018(3)
C(30)	2a	0.2953(4)	1.1213(4)	0.1717(2)	0.052(2)	0.046(3)	0.079(3)	0.001(2)	0.015(2)	0.009(2)
C(31)	2a	0.2060(9)	1.0730(7)	0.1206(4)	0.135(6)	0.069(4)	0.116(5)	0.010(4)	-0.012(4)	0.003(4)
C(32)	2a	0.234(1)	0.967(1)	0.0811(5)	0.22(1)	0.088(5)	0.130(6)	-0.013(8)	0.019(7)	-0.010(5)
C(33)	2a	0.353(1)	0.913(1)	0.0933(5)	0.153(8)	0.103(7)	0.152(7)	0.056(7)	0.042(6)	0.037(6)
C(34)	2a	0.4476(9)	0.9483(7)	0.1434(7)	0.101(5)	0.058(4)	0.29(1)	-0.002(4)	0.110(7)	0.012(6)
C(35)	2a	0.4117(7)	1.0626(7)	0.1871(7)	0.068(4)	0.065(4)	0.29(1)	0.001(4)	0.012(5)	0.050(6)
O(51)	2a	0.7784(5)	1.0466(5)	-0.0345(2)	0.120(3)	0.104(3)	0.089(2)	-0.025(3)	-0.001(2)	-0.004(3)
C(52)	2a	0.8802(8)	1.1371(9)	-0.0241(4)	0.118(5)	0.114(6)	0.098(4)	-0.024(5)	0.010(4)	0.012(4)
C(53)	2a	0.864(1)	1.2036(8)	0.0400(4)	0.149(7)	0.082(5)	0.114(5)	-0.017(5)	-0.018(5)	-0.003(5)
O(54)	2a	0.8707(6)	1.1238(7)	0.0938(2)	0.122(4)	0.149(6)	0.092(3)	-0.024(4)	0.000(3)	-0.016(3)
C(55)	2a	0.7630(8)	1.038(1)	0.0816(4)	0.108(5)	0.172(9)	0.105(5)	-0.030(6)	0.007(4)	0.008(6)
C(56)	2a	0.774(1)	0.966(1)	0.0197(4)	0.172(8)	0.109(6)	0.104(5)	-0.036(7)	-0.003(5)	-0.007(5)
O(61)	2a	0.3037(5)	0.5305(8)	0.4996(3)	0.113(4)	0.191(7)	0.148(5)	0.060(5)	0.022(3)	0.087(5)
C(62)	2a	0.397(1)	0.556(2)	0.4534(5)	0.19(1)	0.28(2)	0.116(6)	0.09(1)	0.048(7)	0.11(1)
C(63)	2a	0.525(1)	0.574(3)	0.480(2)	0.12(1)	0.37(4)	0.64(5)	0.05(2)	0.14(2)	0.32(4)
O(64)	2a	0.5615(8)	0.468(2)	0.5189(7)	0.102(5)	0.36(2)	0.32(1)	0.074(9)	0.003(6)	0.05(2)
C(65)	2a	0.474(1)	0.442(2)	0.5690(5)	0.18(1)	0.23(2)	0.130(7)	0.08(1)	-0.042(7)	0.038(9)
C(66)	2a	0.346(1)	0.437(2)	0.5413(6)	0.139(8)	0.24(2)	0.169(9)	0.05(1)	0.053(7)	0.11(1)

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