Crystal structure of 11-methyl-7,11-diazatricyclo[7.3.1.0^{2,7}]tridecan-13-ols, C₁₂H₂₂N₂O, solid state conformation of *syn*- and *trans*-isomers

H. J. Lindner^I, S. Foro^{*I}, P. Nemes^{II} and P. Scheiber^{II}

^I Technische Universität Darmstadt, Institut für Organische Chemie, Petersenstr. 22, D-64287 Darmstadt, Germany

^{II} University of Veterinary Science, Department of Chemistry, Budapest, Hungary

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Abstract

C₁₂H₂₂N₂O, monoclinic, C12/c1 (No. 15), $a = 21.125(3)$ Å, $b = 14.102(2)$ Å, $c = 20.172(4)$ Å, $\beta = 126.45(1)^\circ$, $V = 4833.9$ Å³, $Z = 16$, $R_{\text{gt}}(F) = 0.054$, $wR_{\text{obs}}(F^2) = 0.142$, $T = 298$ K.

C₁₂H₂₂N₂O, monoclinic, P12₁/c1 (No. 14), $a = 12.527(3)$ Å, $b = 11.171(5)$ Å, $c = 17.757(4)$ Å, $\beta = 106.20(2)^\circ$, $V = 2386.3$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.053$, $wR_{\text{obs}}(F^2) = 0.118$, $T = 300$ K.

Source of material

The substances were prepared as described previously [1]. Colorless prisms of *anti*-11-methyl-7,11-diazatricyclo [3.3.1.0^{2,7}]tridecan-13-ol (α -isomer) were grown by slow recrystallisation from butanone solution at ambient temperature (mp 432 K). Colorless needles of *syn*-11-methyl-7,11-diazatricyclo [7.3.1.0^{2,7}]tridecan-13-ol (β -isomer) were obtained from acetone (mp 418 K).

Experimental details

In the α -isomer all hydrogen atoms were located in difference Fourier maps and refined isotropically, in the β -isomer the hydrogen atoms of the hydroxy groups were found in a difference Fourier map and refined isotropically, the other hydrogen atoms were positioned geometrically.

Discussion

Owing to the easy chair-boat interconversion of the six-membered saturated rings and the repulsion between their *endo* groups/atoms at the 3 and 7 positions the conformation of the bicyclo [3.3.1]nonanes has attracted considerable interest. It seems reasonable to expect the so-called *chair-chair* conformation to be preferred to either the *boat-chair* or *boat-boat* conformation. While the carbocyclic compound [2] and the 3-aza analogue [3] adopt a *chair-chair* conformation, among the 3,7-dihetera (N, S, Se) analogues [4–7] also *chair-boat* forms can occur in crystalline state reflecting the strength of the transannular 3/7 repulsion. Due to the effects on the cardiovascular system [8] the 3,7-diazabicyclo[3.3.1]nonanes were particularly thoroughly studied. Their bicyclic unit constitutes the B and C rings of the molecules of the tri- and tetracyclic lupine alkaloids cytisine and sparteine, respectively. The saturated diazatricyclo[7.3.1.0^{2,7}]tridecanes and some other congeners were also

synthesized [1] and characterized [9] to assume an all *chair* or *chair-chair-boat* conformation in solution depending on the nature and steric position of the C(13) substituent and/or the solvent used. Particularly, the epimeric C(13) title compounds have been found to display striking differences in chloroform solution [9]. While cytisine itself including an α -pyridone moiety has been subjected to structure elucidation by X-ray diffraction in three independent works [10–12], all showing ring C with *chair* conformation its saturated derivatives like the title compounds were not investigated yet.

1. *anti*-11-Methyl-7,11-diazatricyclo [3.3.1.0^{2,7}]tridecan-13-ol

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.55 × 0.325 × 0.325 mm
Wavelength:	Mo K α radiation (0.71093 Å)
μ :	0.7 cm ⁻¹
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{\text{max}}$:	45.94°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3465, 3351
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2608
$N(\text{param})_{\text{refined}}$:	447
Programs:	SHELXS-86 [13], SHELXL-93 [14], PLUTON93 [15], ORTEPIII [16]

* Correspondence author

(e-mail: sabine@friedel.st.mw.tu-darmstadt.de)

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(1)	8f	0.263(1)	-0.014(2)	0.624(2)	0.053(7)
H(2)	8f	0.365(1)	0.044(2)	0.628(2)	0.048(6)
H(31)	8f	0.341(2)	-0.155(2)	0.581(2)	0.056(7)
H(32)	8f	0.376(2)	-0.115(2)	0.666(2)	0.069(9)
H(41)	8f	0.488(2)	-0.053(2)	0.678(2)	0.078(9)
H(42)	8f	0.478(2)	-0.163(2)	0.652(2)	0.08(1)
H(51)	8f	0.497(2)	-0.069(2)	0.569(2)	0.09(1)
H(52)	8f	0.412(2)	-0.127(2)	0.514(2)	0.077(9)
H(61)	8f	0.388(2)	0.038(2)	0.467(2)	0.069(8)
H(62)	8f	0.427(2)	0.067(2)	0.559(2)	0.065(8)
H(81)	8f	0.317(2)	0.151(2)	0.511(2)	0.062(8)
H(82)	8f	0.268(1)	0.108(2)	0.417(2)	0.053(7)
H(9)	8f	0.183(2)	0.175(2)	0.442(2)	0.061(7)
H(101)	8f	0.092(2)	0.057(2)	0.399(2)	0.072(9)
H(102)	8f	0.128(2)	0.050(2)	0.349(2)	0.067(8)
H(121)	8f	0.148(2)	-0.072(2)	0.521(2)	0.057(7)
H(122)	8f	0.207(2)	-0.139(2)	0.528(2)	0.062(8)
H(13)	8f	0.167(2)	0.098(2)	0.537(2)	0.059(8)
H(14)	8f	0.265(2)	0.170(2)	0.636(2)	0.07(1)
H(151)	8f	0.051(2)	-0.102(2)	0.386(2)	0.09(1)
H(152)	8f	0.077(2)	-0.102(2)	0.320(2)	0.09(1)
H(153)	8f	0.107(2)	-0.186(3)	0.391(3)	0.12(2)

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(1A)	8f	0.245(2)	-0.327(2)	0.321(2)	0.060(7)
H(2A)	8f	0.353(2)	-0.240(2)	0.372(2)	0.065(8)
H(3A)	8f	0.316(2)	-0.336(2)	0.233(3)	0.09(1)
H(3B)	8f	0.348(2)	-0.387(3)	0.316(2)	0.09(1)
H(4A)	8f	0.468(3)	-0.309(3)	0.377(3)	0.13(1)
H(4B)	8f	0.456(3)	-0.352(4)	0.302(3)	0.16(2)
H(5A)	8f	0.410(3)	-0.224(3)	0.229(3)	0.13(2)
H(5B)	8f	0.488(3)	-0.190(3)	0.307(3)	0.14(2)
H(6A)	8f	0.430(2)	-0.123(3)	0.362(3)	0.10(1)
H(6B)	8f	0.399(3)	-0.076(3)	0.283(3)	0.13(2)
H(8A)	8f	0.322(2)	-0.065(2)	0.348(2)	0.066(8)
H(8B)	8f	0.279(2)	-0.025(2)	0.255(2)	0.064(8)
H(9A)	8f	0.192(2)	-0.046(2)	0.290(2)	0.067(8)
H(10A)	8f	0.135(1)	-0.068(2)	0.146(2)	0.054(7)
H(10B)	8f	0.090(2)	-0.124(3)	0.175(2)	0.10(1)
H(12A)	8f	0.128(2)	-0.313(2)	0.198(2)	0.069(8)
H(12B)	8f	0.188(2)	-0.341(2)	0.179(2)	0.065(8)
H(13A)	8f	0.162(2)	-0.204(2)	0.307(2)	0.061(8)
H(14A)	8f	0.256(2)	-0.133(2)	0.418(2)	0.07(1)
H(15A)	8f	0.041(2)	-0.242(2)	0.070(2)	0.073(9)
H(15B)	8f	0.070(2)	-0.163(2)	0.037(2)	0.071(9)
H(15C)	8f	0.095(2)	-0.282(3)	0.041(3)	0.12(2)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	8f	0.2547(2)	-0.0022(2)	0.5746(2)	0.077(2)	0.039(1)	0.042(1)	-0.000(1)	0.044(1)	0.001(1)
C(2)	8f	0.3318(2)	-0.0086(2)	0.5858(1)	0.057(2)	0.040(1)	0.034(1)	-0.006(1)	0.025(1)	-0.002(1)
C(3)	8f	0.3735(2)	-0.1023(2)	0.6198(2)	0.058(2)	0.056(2)	0.044(2)	0.001(1)	0.021(1)	0.009(1)
C(4)	8f	0.4530(2)	-0.1020(3)	0.6348(2)	0.050(2)	0.068(2)	0.069(2)	0.006(2)	0.020(2)	0.009(2)
C(5)	8f	0.4429(2)	-0.0753(2)	0.5569(2)	0.045(2)	0.080(2)	0.071(2)	0.000(2)	0.029(2)	-0.009(2)
C(6)	8f	0.3975(2)	0.0161(2)	0.5224(2)	0.054(2)	0.061(2)	0.053(2)	-0.012(1)	0.031(1)	-0.010(1)
N(7)	8f	0.3215(1)	0.0118(1)	0.5083(1)	0.051(1)	0.043(1)	0.037(1)	-0.0006(9)	0.0272(9)	-0.0004(8)
C(8)	8f	0.2794(2)	0.1014(2)	0.4717(2)	0.077(2)	0.041(1)	0.045(2)	0.004(1)	0.043(1)	0.007(1)
C(9)	8f	0.2044(2)	0.1104(2)	0.4634(2)	0.077(2)	0.041(1)	0.049(2)	0.016(1)	0.039(1)	0.010(1)
C(10)	8f	0.1404(2)	0.0410(2)	0.4031(2)	0.060(2)	0.070(2)	0.054(2)	0.021(2)	0.031(2)	0.011(2)
N(11)	8f	0.1606(1)	-0.0578(2)	0.4289(1)	0.046(1)	0.054(1)	0.050(1)	0.001(1)	0.026(1)	-0.006(1)
C(12)	8f	0.1905(2)	-0.0729(2)	0.5147(2)	0.061(2)	0.052(2)	0.058(2)	0.003(1)	0.042(1)	0.003(1)
C(13)	8f	0.2196(2)	0.0970(2)	0.5465(2)	0.078(2)	0.045(1)	0.057(2)	0.007(1)	0.049(2)	0.001(1)
O(14)	8f	0.2704(1)	0.1696(1)	0.6012(1)	0.117(2)	0.045(1)	0.065(1)	-0.009(1)	0.070(1)	-0.0123(9)
C(15)	8f	0.0925(2)	-0.1185(3)	0.3753(3)	0.048(2)	0.098(3)	0.078(2)	-0.014(2)	0.033(2)	-0.025(2)
C(1A)	8f	0.2388(2)	-0.2687(2)	0.2916(2)	0.072(2)	0.036(1)	0.041(1)	-0.004(1)	0.037(1)	0.001(1)
C(2A)	8f	0.3187(2)	-0.2495(2)	0.3123(1)	0.055(2)	0.058(2)	0.030(1)	0.009(1)	0.019(1)	-0.002(1)
C(3A)	8f	0.3501(2)	-0.3290(3)	0.2896(2)	0.087(3)	0.107(3)	0.047(2)	0.044(2)	0.028(2)	-0.005(2)
C(4A)	8f	0.4336(3)	-0.3062(7)	0.3180(3)	0.078(3)	0.285(9)	0.055(3)	0.092(5)	0.013(2)	-0.030(4)
C(5A)	8f	0.4347(3)	-0.2129(8)	0.2841(3)	0.041(2)	0.32(1)	0.067(3)	-0.003(4)	0.028(2)	-0.043(5)
C(6A)	8f	0.3990(2)	-0.1368(4)	0.3037(2)	0.058(2)	0.179(5)	0.059(2)	-0.046(3)	0.034(2)	-0.037(3)
N(7A)	8f	0.3193(1)	-0.1610(2)	0.2733(1)	0.047(1)	0.074(2)	0.038(1)	-0.018(1)	0.025(1)	-0.012(1)
C(8A)	8f	0.2849(2)	-0.0809(2)	0.2893(2)	0.095(2)	0.043(2)	0.044(2)	-0.019(1)	0.042(2)	-0.011(1)
C(9A)	8f	0.2071(2)	-0.1024(2)	0.2721(2)	0.073(2)	0.049(2)	0.048(2)	0.012(1)	0.037(1)	-0.003(1)
C(10A)	8f	0.1398(2)	-0.1203(2)	0.1811(2)	0.064(2)	0.070(2)	0.051(2)	0.016(2)	0.030(2)	0.005(2)
N(11A)	8f	0.1524(1)	-0.2039(2)	0.1487(1)	0.046(1)	0.061(1)	0.037(1)	-0.008(1)	0.0216(9)	-0.005(1)
C(12A)	8f	0.1718(2)	-0.2868(2)	0.1999(2)	0.062(2)	0.055(2)	0.054(2)	-0.016(1)	0.036(1)	-0.006(1)
C(13A)	8f	0.2136(2)	-0.1883(2)	0.3203(2)	0.062(2)	0.065(2)	0.046(2)	0.001(1)	0.038(1)	-0.000(1)

2. *syn*-11-Methyl-7,11-diazatricyclo[7.3.1.0^{2,7}]tridecan-13-ol

Table 4. Data collection and handling.

Crystal:	colourless, thin laminar, size 0.025 × 0.275 × 1.0 mm
Wavelength:	Mo K α radiation (0.71093 Å)
μ :	0.7 cm ⁻¹
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{\max}$:	45.96°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3490, 3304
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1034
$N(\text{param})_{\text{refined}}$:	279
Programs:	SHELXS-86 [13], SHELXL-93 [14], PLUTON93 [15], ORTEPIII [16]

Table 5. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.5978(5)	1.1732(5)	0.3156(4)	0.067
H(2)	4e	0.5641(5)	1.0605(6)	0.4152(4)	0.069
H(31)	4e	0.7499(5)	1.0680(7)	0.4175(4)	0.097
H(32)	4e	0.7358(5)	0.9524(7)	0.3656(4)	0.097
H(41)	4e	0.8145(7)	0.8945(9)	0.4934(4)	0.131
H(42)	4e	0.7222(7)	0.9615(9)	0.5215(4)	0.131
H(51)	4e	0.6900(7)	0.7522(8)	0.4288(4)	0.116
H(52)	4e	0.6765(7)	0.7617(8)	0.5138(4)	0.116
H(61)	4e	0.5027(6)	0.7606(6)	0.4193(4)	0.094
H(62)	4e	0.5194(6)	0.8782(6)	0.4697(4)	0.094
H(81)	4e	0.3759(5)	0.9519(5)	0.3645(4)	0.081
H(82)	4e	0.3757(5)	0.8420(5)	0.3098(4)	0.081
H(9)	4e	0.2984(5)	1.0151(5)	0.2389(4)	0.075
H(101)	4e	0.3725(5)	0.8798(6)	0.1674(4)	0.084
H(102)	4e	0.3838(5)	1.0114(6)	0.1400(4)	0.084
H(121)	4e	0.5775(5)	1.1093(5)	0.1909(3)	0.071
H(122)	4e	0.6697(5)	1.0301(5)	0.2465(3)	0.071
H(13)	4e	0.4138(5)	1.1520(5)	0.3249(4)	0.071
H(14)	4e	0.437(8)	1.27(1)	0.232(7)	0.24(6)
H(151)	4e	0.6334(6)	0.8974(6)	0.1355(3)	0.113
H(152)	4e	0.5306(6)	0.9719(6)	0.0879(3)	0.113
H(153)	4e	0.5170(6)	0.8356(6)	0.1043(3)	0.113
H(1A)	4e	0.0994(5)	0.5691(5)	0.8231(3)	0.060
H(2A)	4e	0.0749(4)	0.6855(5)	0.9245(3)	0.062
H(3A)	4e	0.2571(5)	0.6856(6)	0.9146(4)	0.082
H(3B)	4e	0.2313(5)	0.7997(6)	0.8610(4)	0.082
H(4A)	4e	0.3212(6)	0.8601(6)	0.9874(4)	0.104
H(4B)	4e	0.2331(6)	0.7936(6)	1.0203(4)	0.104
H(5A)	4e	0.1844(6)	0.9952(6)	1.0077(4)	0.093
H(5B)	4e	0.1884(6)	0.9963(6)	0.9201(4)	0.093
H(6A)	4e	0.0305(5)	0.8720(5)	0.9762(3)	0.073
H(6B)	4e	0.0049(5)	0.9849(5)	0.9216(3)	0.073
H(8A)	4e	-0.1323(5)	0.8889(5)	0.8255(4)	0.072
H(8B)	4e	-0.1164(5)	0.7807(5)	0.8839(4)	0.072
H(9A)	4e	-0.2072(5)	0.7112(5)	0.7617(4)	0.068
H(10A)	4e	-0.1398(5)	0.7161(5)	0.6543(4)	0.074
H(10B)	4e	-0.1500(5)	0.8481(5)	0.6814(4)	0.074
H(12A)	4e	0.1542(5)	0.7129(6)	0.7447(3)	0.074
H(12B)	4e	0.0623(5)	0.6268(6)	0.6954(3)	0.074
H(13A)	4e	-0.0788(5)	0.5810(5)	0.8426(4)	0.070
H(14A)	4e	-0.049(7)	0.46(1)	0.735(6)	0.23(5)
H(15A)	4e	0.0974(6)	0.8339(6)	0.6286(3)	0.102
H(15B)	4e	-0.0215(6)	0.8913(6)	0.6040(3)	0.102
H(15C)	4e	-0.0066(6)	0.7546(6)	0.5898(3)	0.102

Table 6. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	4e	0.5591(5)	1.0967(5)	0.3014(4)	0.055(4)	0.031(4)	0.084(5)	-0.014(3)	0.023(4)	-0.008(4)
C(2)	4e	0.5875(5)	1.0178(6)	0.3743(4)	0.066(5)	0.048(5)	0.064(5)	-0.004(4)	0.025(4)	-0.016(4)
C(3)	4e	0.7101(5)	0.9928(7)	0.4057(4)	0.071(5)	0.082(5)	0.085(5)	0.001(4)	0.012(4)	-0.013(5)
C(4)	4e	0.7364(7)	0.9162(9)	0.4787(4)	0.117(7)	0.127(9)	0.069(6)	0.028(7)	0.004(6)	0.000(6)
C(5)	4e	0.6662(7)	0.8043(8)	0.4647(4)	0.134(8)	0.082(6)	0.071(6)	0.022(6)	0.022(6)	0.021(5)
C(6)	4e	0.5454(6)	0.8341(6)	0.4311(4)	0.110(7)	0.059(5)	0.074(5)	0.009(5)	0.041(5)	0.009(4)
N(7)	4e	0.5271(4)	0.9054(4)	0.3600(3)	0.057(4)	0.040(4)	0.067(4)	0.002(3)	0.024(3)	0.003(3)
C(8)	4e	0.4085(5)	0.9200(5)	0.3252(4)	0.073(5)	0.040(4)	0.101(6)	0.000(4)	0.044(5)	0.006(4)
C(9)	4e	0.3790(5)	1.0023(5)	0.2539(4)	0.049(4)	0.030(4)	0.110(6)	-0.001(3)	0.025(4)	0.009(4)
C(10)	4e	0.4098(5)	0.9556(6)	0.1830(4)	0.069(5)	0.036(4)	0.093(6)	-0.002(4)	0.003(4)	0.009(4)
N(11)	4e	0.5286(4)	0.9387(4)	0.1976(3)	0.064(4)	0.033(3)	0.063(4)	0.004(3)	0.016(3)	-0.002(3)
C(12)	4e	0.5908(5)	1.0480(5)	0.2311(3)	0.063(4)	0.039(4)	0.077(5)	-0.003(4)	0.024(4)	0.007(4)
C(13)	4e	0.4348(5)	1.1218(5)	0.2792(4)	0.061(5)	0.031(4)	0.088(5)	0.003(3)	0.023(4)	0.006(4)
O(14)	4e	0.3980(4)	1.2029(5)	0.2151(3)	0.082(3)	0.035(3)	0.133(5)	0.005(3)	0.030(3)	0.018(3)
C(15)	4e	0.5546(6)	0.9083(6)	0.1252(3)	0.153(7)	0.065(5)	0.069(5)	0.014(5)	0.040(5)	0.009(4)
C(1A)	4e	0.0565(5)	0.6431(5)	0.8093(3)	0.063(4)	0.024(3)	0.066(4)	0.017(3)	0.023(4)	-0.004(3)
C(2A)	4e	0.0910(4)	0.7264(5)	0.8800(3)	0.055(4)	0.034(4)	0.070(5)	0.011(3)	0.024(4)	-0.002(3)
C(3A)	4e	0.2133(5)	0.7585(6)	0.9040(4)	0.049(4)	0.069(5)	0.083(5)	0.016(4)	0.009(4)	-0.001(4)
C(4A)	4e	0.2437(6)	0.8369(6)	0.9756(4)	0.093(6)	0.075(6)	0.078(6)	-0.008(5)	0.001(5)	-0.021(4)
C(5A)	4e	0.1704(6)	0.9478(6)	0.9602(4)	0.111(6)	0.053(5)	0.061(5)	-0.002(5)	0.012(5)	-0.013(4)

Table 6. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(6A)	4e	0.0500(5)	0.9131(5)	0.9338(3)	0.085(5)	0.041(4)	0.062(4)	0.011(4)	0.031(4)	-0.006(4)
N(7A)	4e	0.0249(4)	0.8358(4)	0.8651(3)	0.049(3)	0.033(3)	0.054(3)	0.002(3)	0.016(3)	-0.008(3)
C(8A)	4e	-0.0936(5)	0.8135(5)	0.8402(4)	0.054(4)	0.046(4)	0.086(5)	0.010(3)	0.030(4)	-0.001(4)
C(9A)	4e	-0.1276(5)	0.7277(5)	0.7714(4)	0.047(4)	0.045(4)	0.075(5)	-0.001(3)	0.011(4)	-0.007(4)
C(10A)	4e	-0.1096(5)	0.7736(5)	0.6957(4)	0.068(5)	0.031(4)	0.077(5)	-0.001(3)	0.006(4)	-0.008(4)
N(11A)	4e	0.0072(4)	0.7939(4)	0.7011(3)	0.067(4)	0.043(3)	0.045(3)	-0.001(3)	0.016(3)	-0.001(3)
C(12A)	4e	0.0767(5)	0.6904(6)	0.7341(3)	0.066(4)	0.056(5)	0.068(5)	-0.002(4)	0.026(4)	-0.020(4)
C(13A)	4e	-0.0659(5)	0.6109(5)	0.7941(4)	0.071(5)	0.034(4)	0.073(5)	-0.002(4)	0.026(4)	-0.003(4)
O(14A)	4e	-0.1059(4)	0.5259(4)	0.7334(3)	0.086(3)	0.034(3)	0.100(4)	-0.005(3)	0.014(3)	-0.016(3)

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