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Crystal structure of taxuspinanane A acetonitrile solvate, $C_{47}H_{59}NO_{14} \cdot CH_3CN$

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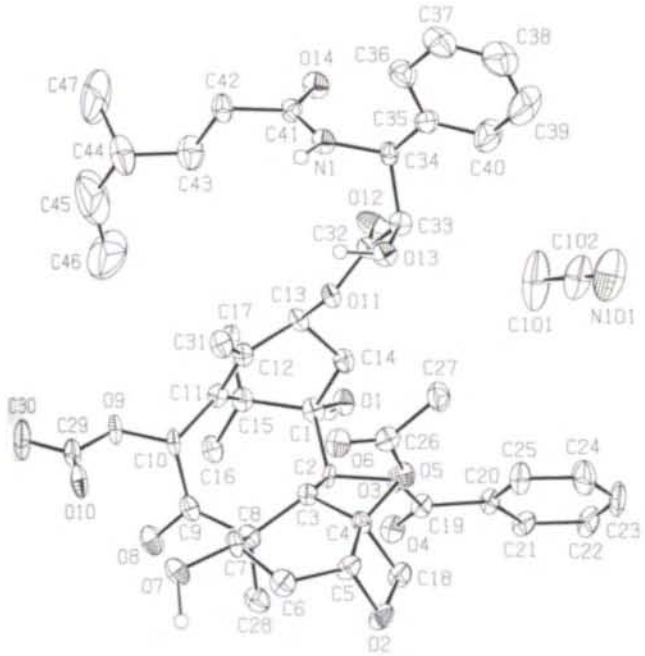

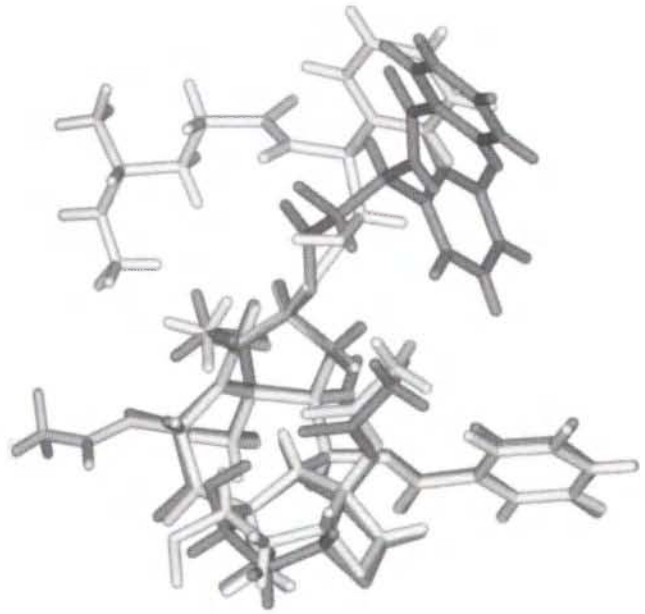

Abstract

$C_{49}H_{62}N_2O_{14}$, orthorhombic, $P2_12_12_1$ (no. 19), a = 9.46370(3) Å, b = 12.29468(3) Å, c = 40.46176(4) A, V = 4707.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.102, $wR_{ref}(F)$ = 0.099, T = 150 K.

Source of material

Mixture of taxanes was isolated by extraction of the stems of *Taxus canadensis* with methanol. Pure taxuspinanane A was obtained by a combination of chromatography on a silica gel (Merck) using the mixture of ethyl acetate/toluene (4:6, *v/v*), and preparative chromatography on a column (21.2 × 250 mm, I.D.) filled with a silica gel (Phenomenex, Luna Silica, 5 μm) using the mixture of 4-methyl-2-pentanone/toluene (4:6, *v/v*). Identity of sample was verified by MS and NMR according to published data [1]. Single crystals of taxuspinanane A were obtained by the slow evaporation of its solution in acetonitrile (30 mg/3 ml) at ambient temperature (298 K).

Discussion

The discovery of paclitaxel as a potent acticancer drug isolated originally from *Taxus brevifolia* has stimulated the isolation of roughly 400 natural taxanes from various species of *Taxus* [2,3]. However, despite the fact that the oligocyclic skeleton of taxanes is rigid, crystal structure determinations of taxanes are rather rare. Apparently, it is facilitated by the fact that majority of taxanes crystallize in the form of fibrous needles, which are not suitable for the X-ray diffraction. Taxuspinanane A is, besides paclitaxel [4] and 10-deacetyl-epipaclitaxel [5], the third crystal structure of a natural taxoid with an oxetane ring and a phenylisoserine C-13 side chain reported so far. Taxuspinanane A, originally obtained from *Taxus cuspidata var. nana* [1], is reported here from *Taxus canadensis* as the 9th paclitaxel analogue extracted from this species [6-8]. It is worth mentioning that taxuspinanane A exhibits about three times higher cell growth inhibitory activity than paclitaxel itself [1]. Taxuspinanane A was crystallized as an acetonitrile solvate. The solvent occupies a distinct area, which does not form channels. Despite this fact, rapid desolvation to an anhydrate even at low temperature is likely the source of lower quality of a single crystal and the large $R_{gt}(F)$ value.

The most remarkable property of taxuspinanane A is the presence of (*S*)-4-methyl-hexanoic acid moiety. Apparently, no crystal structure containing this acid was reported yet. The acid resembles homoisoleucine, an unusual amino acid reported in ergot alkaloid ergogaline and possesses the identical chirality [9]. The conformation of taxuspinanane A backbone is essentially similar to the conformation of baccatin III [10], paclitaxel [4], and some of its semisynthetic derivatives [11-14]. However, in contrast to all structures reported so far, just 2-hydroxy-3-(4-methyl-hexano-

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ylamino)-3-phenyl-propionyl C13 side chain in taxuspinanane A possesses quite different orientation even in comparison with structure of closely related cephalomannine derivative [15]. The conformation of C13 side chain brings the 4-methyl-hexanoyl chain to the close proximity of 10-acetyl group. For example, figure, bottom, shows the comparison of taxuspinanane A (gray) with one of two crystallographically independent molecules of 10-deacetyl-7-epipaclitaxel (black) [5]. Concerning the H bond network, hydrogen atoms bonded to O13 and N1 form both a H bond with x+0.5,–y+0.5,–z–2 related O14. O1 forms a H bond with x–1,y,z related O7. The network of H bonds creates a system of 2D layers. Individual layers are hold together to 3D structure by weak interactions only.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.16 × 0.23 × 0.30 mm
Wavelength:	Cu K_α radiation (1.54180 Å)
μ:	7.69 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Xcalibur, φ/ω
$2\theta_{max}$:	153.66°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	72545, 9646
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 9105
$N(param)_{refined}$:	587
Programs:	SIR92 [16], CRYSTALS [17], CAMERON [18]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	–0.9418	0.2020	–0.8109	0.029
H(2)	4*a*	–0.0568	0.2097	–0.7751	0.037
H(3)	4*a*	–0.4064	0.0927	–0.9756	0.032
H(11)	4*a*	–0.5100	0.2125	–1.0117	0.029
H(21)	4*a*	–0.6392	0.1497	–0.7908	0.021
H(31)	4*a*	–0.4536	0.1375	–0.8415	0.024
H(51)	4*a*	–0.1958	–0.0693	–0.8266	0.031
H(61)	4*a*	–0.0695	0.0455	–0.8145	0.037
H(62)	4*a*	–0.1368	0.0538	–0.7774	0.037
H(71)	4*a*	–0.2241	0.1773	–0.8338	0.029
H(101)	4*a*	–0.3344	0.3310	–0.8471	0.026
H(131)	4*a*	–0.7118	0.2598	–0.9071	0.024
H(141)	4*a*	–0.8090	0.1159	–0.8810	0.030
H(142)	4*a*	–0.6556	0.0631	–0.8719	0.030
H(161)	4*a*	–0.6748	0.4270	–0.8012	0.031
H(162)	4*a*	–0.7955	0.3368	–0.7923	0.031
H(163)	4*a*	–0.6287	0.3083	–0.7867	0.031
H(171)	4*a*	–0.7746	0.4475	–0.8549	0.030
H(172)	4*a*	–0.8016	0.3443	–0.8795	0.030
H(173)	4*a*	–0.8942	0.3568	–0.8456	0.030
H(181)	4*a*	–0.4950	–0.1559	–0.7951	0.033
H(182)	4*a*	–0.5067	–0.0492	–0.7712	0.033
H(211)	4*a*	–0.9751	–0.1396	–0.7587	0.039
H(221)	4*a*	–1.0621	–0.3202	–0.7689	0.047
H(231)	4*a*	–0.9646	–0.4259	–0.8120	0.056
H(241)	4*a*	–0.7871	–0.3535	–0.8472	0.056
H(251)	4*a*	–0.7029	–0.1727	–0.8385	0.043
H(271)	4*a*	–0.3682	–0.0996	–0.9241	0.045
H(272)	4*a*	–0.5272	–0.1100	–0.9087	0.045
H(273)	4*a*	–0.4046	–0.1963	–0.8977	0.045
H(281)	4*a*	–0.3201	0.2030	–0.7465	0.037
H(282)	4*a*	–0.4820	0.1599	–0.7514	0.037
H(283)	4*a*	–0.3497	0.0757	–0.7557	0.037
H(301)	4*a*	–0.2304	0.6753	–0.8160	0.066
H(302)	4*a*	–0.3656	0.6723	–0.8411	0.066
H(303)	4*a*	–0.3891	0.6545	–0.8014	0.066
H(311)	4*a*	–0.4004	0.2451	–0.9252	0.034
H(312)	4*a*	–0.3660	0.3611	–0.9072	0.034
H(313)	4*a*	–0.3097	0.2477	–0.8908	0.034
H(331)	4*a*	–0.6550	–0.0356	–0.9732	0.030
H(341)	4*a*	–0.7715	0.0904	–1.0086	0.025
H(361)	4*a*	–0.5745	0.1451	–1.0698	0.047
H(371)	4*a*	–0.5307	0.0329	–1.1170	0.055
H(381)	4*a*	–0.5580	–0.1578	–1.1136	0.069
H(391)	4*a*	–0.6356	–0.2380	–1.0631	0.095
H(401)	4*a*	–0.6866	–0.1236	–1.0163	0.070
H(421)	4*a*	–0.6736	0.4451	–1.0296	0.037
H(422)	4*a*	–0.5269	0.3882	–1.0172	0.037
H(431)	4*a*	–0.7510	0.4613	–0.9746	0.057
H(432)	4*a*	–0.6097	0.3983	–0.9616	0.057
H(441)	4*a*	–0.4698	0.5386	–0.9791	0.083
H(451)	4*a*	–0.6917	0.6357	–0.9425	0.166
H(452)	4*a*	–0.5287	0.6791	–0.9429	0.166
H(461)	4*a*	–0.5939	0.6150	–0.8949	0.184
H(462)	4*a*	–0.6337	0.4991	–0.9123	0.184
H(463)	4*a*	–0.4707	0.5426	–0.9127	0.184
H(471)	4*a*	–0.5711	0.7080	–0.9955	0.310
H(472)	4*a*	–0.6094	0.6105	–1.0214	0.310
H(473)	4*a*	–0.7276	0.6521	–0.9949	0.310
H(1011)	4*a*	–1.0832	–0.1063	–0.8992	0.106
H(1012)	4*a*	–0.9997	0.0068	–0.8911	0.106
H(1013)	4*a*	–0.9837	–0.0973	–0.8663	0.106

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	–0.8681(3)	0.1598(2)	–0.82897(7)	0.011(1)	0.024(1)	0.037(2)	–0.002(1)	0.003(1)	–0.002(1)
O(2)	4*a*	–0.3021(3)	–0.0975(3)	–0.77976(8)	0.023(2)	0.032(2)	0.041(2)	–0.008(1)	0.001(1)	0.015(1)
O(3)	4*a*	–0.6854(3)	0.0024(2)	–0.81436(7)	0.015(1)	0.017(1)	0.030(1)	–0.008(1)	0.006(1)	0.002(1)
O(4)	4*a*	–0.8169(3)	0.0269(2)	–0.76790(7)	0.021(1)	0.029(2)	0.034(2)	–0.001(1)	0.008(1)	0.000(1)
O(5)	4*a*	–0.4556(3)	–0.0692(2)	–0.84819(7)	0.017(1)	0.024(1)	0.028(1)	–0.004(1)	0.003(1)	–0.004(1)
O(6)	4*a*	–0.2901(3)	0.0213(3)	–0.87711(8)	0.026(2)	0.032(2)	0.036(2)	–0.002(1)	0.008(1)	–0.001(1)
O(7)	4*a*	–0.1218(3)	0.2447(3)	–0.79545(8)	0.014(1)	0.039(2)	0.037(2)	–0.007(1)	–0.003(1)	–0.005(1)
O(8)	4*a*	–0.3856(3)	0.3636(3)	–0.77606(8)	0.027(2)	0.032(2)	0.034(2)	–0.002(1)	0.004(1)	–0.013(1)
O(9)	4*a*	–0.4269(3)	0.4714(2)	–0.83072(8)	0.030(2)	0.016(1)	0.041(2)	–0.010(1)	0.002(1)	–0.007(1)
O(10)	4*a*	–0.1915(3)	0.4734(3)	–0.82314(9)	0.033(2)	0.029(2)	0.051(2)	–0.017(2)	0.002(2)	–0.009(1)
O(11)	4*a*	–0.5847(3)	0.1407(2)	–0.92533(7)	0.019(1)	0.022(1)	0.026(1)	–0.004(1)	0.000(1)	–0.006(1)
O(12)	4*a*	–0.8043(3)	0.1334(3)	–0.94680(8)	0.021(2)	0.059(2)	0.031(2)	0.008(2)	–0.003(1)	–0.015(2)
O(13)	4*a*	–0.4707(3)	0.0259(2)	–0.97347(7)	0.017(1)	0.029(2)	0.034(2)	0.006(1)	–0.001(1)	–0.003(1)
O(14)	4*a*	–0.8298(3)	0.2795(3)	–1.01610(8)	0.014(1)	0.035(2)	0.042(2)	0.002(1)	–0.003(1)	0.000(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4a	−0.6144(3)	0.1999(3)	−1.01139(9)	0.011(1)	0.027(2)	0.034(2)	0.001(1)	0.001(1)	−0.001(2)
N(101)	4a	−0.7822(6)	−0.1413(5)	−0.9225(2)	0.051(3)	0.050(3)	0.127(5)	0.000(3)	0.033(4)	−0.007(3)
C(1)	4a	−0.7231(4)	0.1784(3)	−0.8377(1)	0.007(2)	0.024(2)	0.029(2)	0.003(1)	0.005(1)	−0.005(2)
C(2)	4a	−0.6312(4)	0.1117(3)	−0.8126(1)	0.018(2)	0.008(2)	0.028(2)	−0.003(1)	0.003(2)	−0.000(1)
C(3)	4a	−0.4661(4)	0.1065(3)	−0.8189(1)	0.015(2)	0.019(2)	0.025(2)	0.001(1)	0.001(2)	−0.001(2)
C(4)	4a	−0.4134(4)	−0.0121(3)	−0.8180(1)	0.016(2)	0.021(2)	0.026(2)	−0.002(2)	−0.002(2)	−0.003(2)
C(5)	4a	−0.2565(4)	−0.0368(4)	−0.8090(1)	0.013(2)	0.030(2)	0.035(2)	−0.001(2)	0.005(2)	0.011(2)
C(6)	4a	−0.1586(4)	0.0560(4)	−0.8016(1)	0.015(2)	0.036(2)	0.041(2)	−0.002(2)	0.001(2)	0.003(2)
C(7)	4a	−0.2173(4)	0.1664(3)	−0.8093(1)	0.015(2)	0.023(2)	0.034(2)	−0.007(2)	0.001(2)	−0.005(2)
C(8)	4a	−0.3733(4)	0.1801(3)	−0.7962(1)	0.017(2)	0.028(2)	0.027(2)	−0.004(2)	0.000(2)	−0.003(2)
C(9)	4a	−0.3994(4)	0.3030(3)	−0.7998(1)	0.010(2)	0.027(2)	0.032(2)	−0.004(2)	0.001(2)	−0.006(2)
C(10)	4a	−0.4188(4)	0.3543(3)	−0.8341(1)	0.020(2)	0.013(2)	0.033(2)	−0.007(1)	0.003(2)	−0.006(2)
C(11)	4a	−0.5429(4)	0.3145(3)	−0.8531(1)	0.008(2)	0.019(2)	0.031(2)	0.003(1)	0.002(2)	0.001(2)
C(12)	4a	−0.5233(4)	0.2724(3)	−0.8839(1)	0.021(2)	0.020(2)	0.027(2)	−0.001(2)	0.000(2)	−0.002(2)
C(13)	4a	−0.6395(4)	0.2073(3)	−0.89865(9)	0.013(2)	0.023(2)	0.025(2)	0.000(2)	0.001(1)	−0.008(2)
C(14)	4a	−0.7113(4)	0.1321(3)	−0.87306(9)	0.023(2)	0.024(2)	0.026(2)	−0.004(2)	0.001(2)	−0.003(2)
C(15)	4a	−0.6878(4)	0.3027(3)	−0.8367(1)	0.021(2)	0.022(2)	0.029(2)	0.001(2)	0.002(2)	−0.002(2)
C(16)	4a	−0.6977(4)	0.3476(3)	−0.8011(1)	0.018(2)	0.027(2)	0.034(2)	−0.004(2)	0.005(2)	−0.007(2)
C(17)	4a	−0.7993(4)	0.3685(3)	−0.8559(1)	0.014(2)	0.017(2)	0.043(2)	0.004(2)	0.001(2)	0.001(2)
C(18)	4a	−0.4487(4)	−0.0860(4)	−0.7886(1)	0.019(2)	0.026(2)	0.037(2)	−0.001(2)	0.001(2)	0.006(2)
C(19)	4a	−0.7801(4)	−0.0292(3)	−0.7912(1)	0.005(2)	0.025(2)	0.033(2)	−0.004(1)	0.004(1)	0.006(2)
C(20)	4a	−0.8345(4)	−0.1398(3)	−0.7982(1)	0.006(2)	0.022(2)	0.041(2)	−0.006(1)	−0.001(2)	0.006(2)
C(21)	4a	−0.9369(4)	−0.1833(4)	−0.7776(1)	0.018(2)	0.033(2)	0.042(2)	−0.013(2)	−0.004(2)	0.017(2)
C(22)	4a	−0.9858(5)	−0.2891(4)	−0.7831(1)	0.020(2)	0.032(2)	0.066(3)	−0.016(2)	−0.005(2)	0.018(2)
C(23)	4a	−0.9297(6)	−0.3501(4)	−0.8084(2)	0.040(3)	0.019(2)	0.079(4)	−0.015(2)	−0.011(3)	0.005(2)
C(24)	4a	−0.8267(6)	−0.3079(4)	−0.8289(2)	0.035(3)	0.038(3)	0.065(4)	−0.015(2)	0.000(2)	−0.002(3)
C(25)	4a	−0.7784(5)	−0.2033(4)	−0.8239(1)	0.031(2)	0.021(2)	0.053(3)	−0.002(2)	0.004(2)	−0.006(2)
C(26)	4a	−0.3816(5)	−0.0457(4)	−0.8760(1)	0.028(2)	0.027(2)	0.027(2)	0.008(2)	0.005(2)	−0.000(2)
C(27)	4a	−0.4241(6)	−0.1190(4)	−0.9040(1)	0.043(3)	0.026(2)	0.038(3)	−0.004(2)	0.001(2)	−0.007(2)
C(28)	4a	−0.3822(5)	0.1523(4)	−0.7592(1)	0.025(2)	0.039(2)	0.028(2)	−0.009(2)	0.003(2)	−0.002(2)
C(29)	4a	−0.3046(5)	0.5227(4)	−0.8246(1)	0.027(2)	0.024(2)	0.042(2)	−0.009(2)	−0.001(2)	−0.004(2)
C(30)	4a	−0.3241(7)	0.6407(4)	−0.8205(2)	0.050(3)	0.024(2)	0.089(4)	−0.008(2)	−0.004(3)	−0.008(3)
C(31)	4a	−0.3885(4)	0.2826(4)	−0.9034(1)	0.016(2)	0.033(2)	0.034(2)	−0.007(2)	0.004(2)	−0.002(2)
C(32)	4a	−0.6814(4)	0.1090(3)	−0.9477(1)	0.025(2)	0.024(2)	0.024(2)	−0.003(2)	−0.003(2)	−0.001(2)
C(33)	4a	−0.6184(4)	0.0403(3)	−0.9755(1)	0.019(2)	0.025(2)	0.028(2)	0.002(2)	0.002(2)	−0.001(2)
C(34)	4a	−0.6658(4)	0.0887(3)	−1.0086(1)	0.016(2)	0.019(2)	0.028(2)	−0.002(2)	−0.000(2)	−0.001(2)
C(35)	4a	−0.6279(5)	0.0189(4)	−1.0384(1)	0.035(2)	0.026(2)	0.025(2)	0.005(2)	−0.002(2)	−0.002(2)
C(36)	4a	−0.5866(6)	0.0645(4)	−1.0680(1)	0.043(3)	0.043(3)	0.031(2)	−0.008(2)	0.002(2)	−0.005(2)
C(37)	4a	−0.5618(6)	−0.0010(5)	−1.0957(1)	0.046(3)	0.064(4)	0.030(2)	−0.010(3)	0.010(2)	−0.010(2)
C(38)	4a	−0.5775(8)	−0.1116(6)	−1.0937(2)	0.079(5)	0.060(4)	0.042(3)	0.012(3)	0.007(3)	−0.022(3)
C(39)	4a	−0.622(1)	−0.1575(5)	−1.0646(2)	0.19(1)	0.023(3)	0.048(4)	0.019(4)	0.011(5)	−0.013(2)
C(40)	4a	−0.6504(9)	−0.0908(4)	−1.0372(1)	0.112(6)	0.030(3)	0.037(3)	−0.001(3)	0.006(3)	−0.005(2)
C(41)	4a	−0.7001(4)	0.2868(3)	−1.0136(1)	0.018(2)	0.025(2)	0.029(2)	−0.005(2)	0.000(2)	0.002(2)
C(42)	4a	−0.6299(5)	0.3970(4)	−1.0124(1)	0.019(2)	0.025(2)	0.045(3)	−0.003(2)	−0.001(2)	−0.004(2)
C(43)	4a	−0.6479(6)	0.4492(4)	−0.9787(1)	0.050(3)	0.036(3)	0.051(3)	−0.002(2)	0.005(3)	−0.008(2)
C(44)	4a	−0.5709(9)	0.5591(5)	−0.9756(2)	0.082(5)	0.044(3)	0.073(4)	−0.013(3)	−0.010(4)	−0.020(3)
C(45)	4a	−0.590(2)	0.613(1)	−0.9429(3)	0.27(2)	0.100(8)	0.101(8)	−0.06(1)	−0.00(1)	−0.052(7)
C(46)	4a	−0.572(2)	0.5650(9)	−0.9138(2)	0.30(2)	0.088(7)	0.058(5)	−0.04(1)	0.030(8)	0.007(5)
C(47)	4a	−0.625(2)	0.6385(7)	−0.9985(5)	0.42(3)	0.024(4)	0.30(2)	−0.020(9)	−0.21(2)	0.023(7)
C(101)	4a	−0.9946(7)	−0.0743(6)	−0.8899(3)	0.046(4)	0.047(4)	0.171(9)	−0.004(3)	0.028(5)	−0.040(5)
C(102)	4a	−0.8744(6)	−0.1125(5)	−0.9085(2)	0.026(3)	0.035(3)	0.106(5)	−0.001(2)	0.006(3)	−0.010(3)

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