through the atoms C(8), C(9), C(11), C(12), C(15), C(16) and C(17) [maximum deviation 0.081 (9) Å for C(15)] with C(13) 0.394 (7) and C(14) -0.407 (7) Å out of this plane.

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Functionalized Hydrocarbons with Condensed Ring Skeletons. XI. Tetramethyl Tricyclo[8.4.0.0²,⁷]tetradeca-1,8-diene-5,5,12,12-tetracarboxylate and its Corresponding Tetradec-8-ene

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Abstract. Monoclinic, λ(Cu $K\alpha$) = 1.54056 Å, Z = 4, room temperature. (I) (7,10-*cis*)-Tetramethyl tricyclo[8.4.0.0²,⁷]tetradeca-1,8-diene-5,5,12,12-tetracarboxylate, $C_{22}H_{28}O_8$, M_r = 420.46, $P2_1/a$, a = 7.8478 (4), b = 20.0952 (9), c = 13.3666 (3) Å, β = 95.680 (2)°, V = 2097.60 Å³, D_x = 1.331 Mg m⁻³, μ = 0.80 mm⁻¹, $F(000)$ = 896, final R = 0.052 for 2840 observed reflections. (II) (1,2-*cis*-1,10-*trans*-2,7-*trans*)-Tetramethyl tricyclo[8.4.0.0²,⁷]tetradec-8-ene-5,5,12,12-tetracarboxylate, $C_{22}H_{30}O_8$, M_r = 422.48, $P2_1/c$, a = 6.0842 (2), b = 12.7171 (7), c = 27.8011 (16) Å, β = 93.183 (3)°, V = 2147.75 Å³, D_x = 1.307 Mg m⁻³, μ = 0.79 mm⁻¹, $F(000)$ = 904, final R = 0.046 for 3256 observed reflections. Both compounds (I) and (II) consist of three angularly fused six-membered rings A, B and C. Rings A and C for structure (I) adopt a chair conformation with C7 and C10 H atoms (IUPAC numbering) being axial, while unsaturated ring B prefers a boat-like conformation. Rings A and C for structure (II) also adopt a chair conformation with C1, C2, C7 and C10 H atoms (IUPAC numbering) being all axial, while the conformation of ring B is boat. In compound (II), the relative stereochemistry is *trans* at the AB ring junction, *syn* between C1 and C2 H atoms (IUPAC numbering) and *trans* at the BC ring junction (abbreviated by TST).

Introduction. In earlier publications (Michel, Michel-Dewez & Roughton 1989; Drouin, Michel, Xu & Deslongchamps, 1990), we have reported the conformational properties and X-ray data of several steroid-like compounds having $A.B.C.$ [6.6.6] tricyclic skeletons. Two new tricyclic compounds (I) and (II), which do not have the methyl substituent on the ring junctions, were synthesized from their macrocyclic precursors (III) and (IV), respectively (Cantin, Xu & Deslongchamps, 1990). The double bonds $\Delta^{3,4}$ and $\Delta^{5,6}$ are both *trans* in the two macrocycles and $\Delta^{11,12}$ is *cis* in (IV). It is expected that the transannular Diels–Alder reaction takes place *via* a boat-like transition state (Lamothe, Ndibwami & Deslongchamps, 1988). Consequently, the conformational strains imposed on the transition state by its olefin geometry, substituents, and ring size determine the outcome of the relative stereochemistry of the cycloaddition products. From the molecular-model analysis of the transition state, it is expected that 4 + 2 cycloaddition of (III) would give rise to tricyclic product (I) with the two H atoms H(3) and H(6) (crystallographic numbering) on ring B being *syn*. Indeed, only one compound was obtained by the transannular Diels–Alder reaction of (III), but the relative stereochemistry could not be determined by conventional spectroscopic data. From a similar molecular-modeling analysis, the transannular Diels–Alder reaction of macrocycle (IV) would produce a

mixture of tricyclic compounds (II) having *trans*-[H(6),H(11)]-*syn*-[H(11),H(12)-*trans*-[H(12),H(3)] (TST) and *cis-syn-cis* (CSC) stereochemistry. Interestingly, thermolysis of macrocycle (IV) yielded a single product (II). Again, the relative stereochemistry could not be established by spectroscopic data. The crystallographic studies presented herein were undertaken in order to determine the relative stereochemistry of tricyclic compounds (I) and (II).

Transition state
E = COOCH₃

Scheme 1
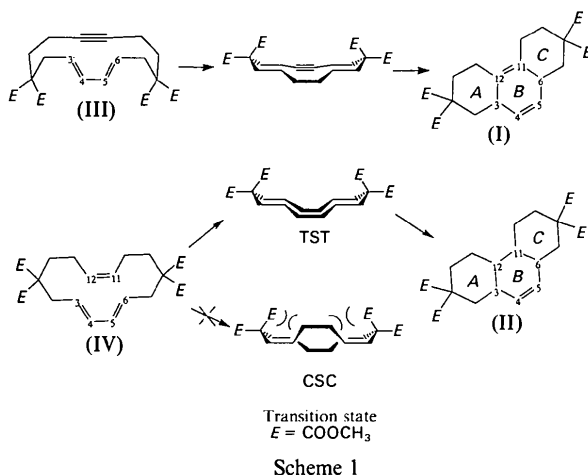
Experimental. An Enraf–Nonius CAD-4 diffractometer was used to collect data with graphite-monochromated Cu *Kα* radiation and 2θ–ω scans. Lattice parameters were determined from 24 medium-angle (60 ≤ 2θ ≤ 100°) reflections. Two standard reflections monitored every 60 min showed no significant deviation.

For compound (I) a crystal of size 0.10 × 0.15 × 0.20 mm was used to collect data for −9 ≤ h ≤ 9, 0 ≤ k ≤ 24, 0 ≤ l ≤ 16 and to 2θ_{max} = 144.3°. Of 4150 unique measured reflections, 2840 were observed with $I_{net} \geq 2.5\sigma I_{net}$. $R = 0.052$, $wR = 0.030$ for 374 parameters; $(\Delta/\sigma)_{max} = 0.20$; maximum and minimum density peaks = 0.370 and −0.370 e Å⁻³.

For (II), crystal size was 0.30 × 0.30 × 0.20 mm and data were collected for −7 ≤ h ≤ 7, 0 ≤ k ≤ 15, 0 ≤ l ≤ 34; 2θ_{max} = 144.3°. Of 4226 unique measured reflections, 3256 were observed with $I_{net} \geq 2.5\sigma I_{net}$. $R = 0.046$, $wR = 0.027$ for 392 parameters; $(\Delta/\sigma)_{max} = 0.15$; maximum and minimum density peaks = 0.240 and −0.180 e Å⁻³.

The secondary-extinction coefficient was 0.50 (2) (Larson, 1967; Zachariasen, 1963). The *NRCVAX* system (Gabe, Lee & Le Page, 1985) was used for all calculations. Both structures were solved by the application of direct methods and refined by full-matrix least squares on *F* using weights based on counting statistics. Weighting scheme $w = 1/(\sigma^2 F)$. No correction was made for absorption. Anisotropic thermal parameters were refined for all non-H atoms

except for C(18) and C(20) in (I) which were refined isotropically because of their high thermal motions. The H atoms were located from a difference map; their positions and isotropic temperature factors were refined. Atomic scattering factors as stored in the *NRCVAX* program are those of Cromer & Waber (1974).

Discussion. Table 1* gives the positional parameters with their B_{eq} values. Fig. 1 shows a perspective view of both compounds with the crystallographic numbering scheme. Bond lengths and angles are given in Table 2. As predicted above, the *trans-trans* diene and acetylene dienophile of (III) allow the macrocycle to take a conformation leading to only one boat-like transition state, which determines the relative stereochemistry of the resulting racemic adduct. The relative stereochemistry of (II) is found to be *trans-syn-trans* (TST). The *trans-trans-cis* macrocycle (IV) can adopt two different conformations which can lead to two tricyclic products with TST and *cis-syn-cis* (CSC) stereochemistry, but the conformation leading to CSC experiences much higher steric energy in the transition state than TST owing to the 1,3 axial steric interactions of the ester
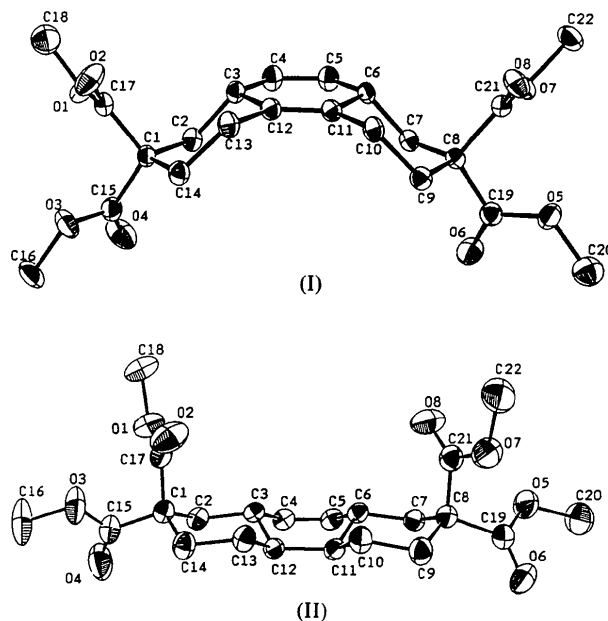
(I)

(II)

Fig. 1. *ORTEP*II (Johnson, 1976) perspective views and crystallographic numbering.

Table 1. *Final coordinates and equivalent isotropic thermal parameters* ($Å^2$) *for* (I) *and* (II)

	x	y	z	B_{eq}
(I)				
C(1)	0.2447 (3)	0.39870 (13)	0.48025 (18)	2.59 (12)
C(2)	0.1853 (4)	0.42935 (15)	0.57562 (21)	2.99 (13)
C(3)	0.1538 (4)	0.37677 (14)	0.65498 (19)	2.80 (13)
C(4)	0.0858 (4)	0.40912 (16)	0.74345 (22)	3.58 (14)
C(5)	0.1568 (4)	0.40206 (16)	0.83669 (21)	3.42 (13)
C(6)	0.3124 (4)	0.36115 (14)	0.86545 (19)	2.60 (12)
C(7)	0.4483 (4)	0.40228 (14)	0.92951 (20)	2.65 (12)
C(8)	0.6081 (3)	0.36191 (13)	0.96424 (18)	2.46 (11)
C(9)	0.6799 (4)	0.32999 (15)	0.87220 (21)	2.89 (12)
C(10)	0.5469 (4)	0.29053 (15)	0.80549 (21)	2.99 (13)
C(11)	0.3869 (3)	0.33004 (12)	0.77566 (18)	2.56 (11)
C(12)	0.3145 (3)	0.33648 (13)	0.68205 (19)	2.69 (12)
C(13)	0.3778 (4)	0.30442 (15)	0.59035 (21)	3.11 (13)
C(14)	0.4025 (4)	0.35441 (15)	0.50715 (20)	3.00 (13)
C(15)	0.2921 (4)	0.45446 (15)	0.41012 (21)	3.37 (14)
C(16)	0.3874 (7)	0.47734 (24)	0.2509 (3)	5.15 (22)
C(17)	0.1004 (4)	0.35839 (15)	0.42385 (19)	3.17 (13)
C(18)	−0.1924 (6)	0.3621 (3)	0.3604 (4)	5.88 (10)
C(19)	0.7493 (4)	0.40720 (14)	1.01210 (20)	3.07 (12)
C(20)	1.0070 (6)	0.40869 (22)	1.1218 (3)	4.94 (8)
C(21)	0.5695 (3)	0.30814 (14)	1.03969 (19)	2.77 (12)
C(22)	0.4540 (6)	0.28779 (20)	1.1936 (3)	4.21 (18)
O(1)	−0.0394 (3)	0.39542 (10)	0.40709 (15)	4.52 (10)
O(2)	0.1091 (3)	0.30202 (11)	0.39740 (16)	5.33 (11)
O(3)	0.3332 (3)	0.42992 (10)	0.32303 (14)	4.67 (12)
O(4)	0.2957 (3)	0.51222 (10)	0.42919 (16)	6.19 (14)
O(5)	0.8520 (3)	0.37467 (10)	1.08031 (15)	4.31 (10)
O(6)	0.7718 (3)	0.46334 (9)	0.98717 (16)	5.05 (11)
O(7)	0.49320 (24)	0.33455 (9)	1.11571 (13)	3.52 (10)
O(8)	0.6040 (3)	0.25041 (9)	1.03395 (13)	3.92 (10)
(II)				
C(1)	0.8720 (4)	0.19403 (15)	0.19110 (7)	2.76 (9)
C(2)	1.0263 (4)	0.20866 (16)	0.14971 (7)	2.85 (9)
C(3)	1.0853 (3)	0.10458 (15)	0.12609 (6)	2.46 (8)
C(4)	1.2361 (3)	0.11760 (16)	0.08547 (7)	2.66 (9)
C(5)	1.2808 (3)	0.03361 (18)	0.05976 (7)	2.82 (9)
C(6)	1.1772 (4)	−0.06970 (15)	0.07173 (6)	2.48 (9)
C(7)	1.2187 (4)	−0.15629 (17)	0.03507 (7)	2.79 (9)
C(8)	1.1038 (4)	−0.26124 (15)	0.04493 (7)	2.75 (9)
C(9)	0.8602 (4)	−0.24310 (18)	0.05475 (8)	3.46 (11)
C(10)	0.8310 (4)	−0.15810 (18)	0.09296 (8)	3.14 (10)
C(11)	0.9283 (4)	−0.05444 (15)	0.07735 (7)	2.54 (9)
C(12)	0.8768 (3)	0.04423 (15)	0.10825 (7)	2.45 (9)
C(13)	0.7322 (4)	0.02392 (18)	0.15041 (7)	2.99 (9)
C(14)	0.6715 (4)	0.12720 (19)	0.17428 (8)	3.32 (10)
C(15)	0.7790 (4)	0.29992 (18)	0.20585 (8)	3.83 (11)
C(16)	0.6174 (9)	0.3906 (3)	0.26986 (16)	7.40 (22)
C(17)	0.9872 (4)	0.14243 (18)	0.23543 (7)	3.47 (10)
C(18)	1.2856 (7)	0.1545 (3)	0.29372 (11)	5.64 (17)
C(19)	1.1184 (4)	−0.33007 (17)	0.00007 (8)	3.53 (10)
C(20)	1.3659 (7)	−0.4225 (3)	−0.04747 (11)	6.43 (19)
C(21)	1.2178 (4)	−0.31924 (16)	0.08735 (7)	3.15 (10)
C(22)	1.2225 (7)	−0.4813 (3)	0.12992 (11)	5.60 (19)
O(1)	1.1716 (3)	0.19244 (12)	0.24884 (5)	4.52 (8)
O(2)	0.9191 (3)	0.06706 (13)	0.25572 (5)	5.54 (9)
O(3)	0.7245 (3)	0.29772 (12)	0.25139 (5)	5.29 (9)
O(4)	0.7486 (3)	0.37350 (13)	0.17981 (6)	6.45 (11)
O(5)	1.3258 (3)	−0.35772 (12)	−0.00550 (5)	4.70 (8)
O(6)	0.9694 (3)	−0.35216 (13)	−0.02779 (5)	5.85 (9)
O(7)	1.1321 (3)	−0.41455 (11)	0.09130 (5)	4.66 (8)
O(8)	1.3607 (3)	−0.28509 (11)	0.11401 (5)	4.98 (8)

Table 2. *Molecular geometry* (Å, °) *for* (I) *and* (II)

	(I)	(II)
C(1)—C(2)	1.529 (4)	1.537 (3)
C(1)—C(14)	1.538 (4)	1.538 (3)
C(1)—C(15)	1.530 (4)	1.526 (3)
C(1)—C(17)	1.529 (4)	1.531 (3)
C(2)—C(3)	1.534 (4)	1.529 (3)
C(3)—C(4)	1.493 (4)	1.503 (3)
C(3)—C(12)	1.512 (4)	1.541 (3)
C(4)—C(5)	1.321 (4)	1.322 (3)
C(5)—C(6)	1.490 (4)	1.502 (3)
C(6)—C(7)	1.540 (4)	1.531 (3)
C(6)—C(11)	1.521 (4)	1.543 (3)
C(7)—C(8)	1.527 (4)	1.538 (3)
C(8)—C(9)	1.543 (4)	1.539 (3)
C(8)—C(19)	1.525 (4)	1.530 (3)
C(8)—C(21)	1.529 (4)	1.525 (3)
C(9)—C(10)	1.526 (4)	1.533 (3)
C(10)—C(11)	1.506 (4)	1.518 (3)
C(11)—C(12)	1.328 (3)	1.562 (3)
C(12)—C(13)	1.512 (4)	1.526 (3)
C(13)—C(14)	1.525 (4)	1.526 (3)
C(15)—O(3)	1.333 (3)	1.327 (3)
C(15)—O(4)	1.188 (4)	1.191 (3)
C(16)—O(3)	1.449 (4)	1.456 (4)
C(17)—O(1)	1.326 (4)	1.325 (3)
C(17)—O(2)	1.191 (4)	1.198 (3)
C(18)—O(1)	1.459 (5)	1.475 (3)
C(19)—O(5)	1.328 (3)	1.328 (3)
C(19)—O(6)	1.194 (3)	1.193 (3)
C(20)—O(5)	1.457 (5)	1.460 (3)
C(21)—O(7)	1.339 (3)	1.326 (3)
C(21)—O(8)	1.195 (3)	1.193 (3)
C(22)—O(7)	1.458 (4)	1.453 (3)
C(2)—C(1)—C(14)	110.05 (21)	110.28 (16)
C(2)—C(1)—C(15)	109.17 (22)	110.22 (17)
C(2)—C(1)—C(17)	110.62 (23)	112.52 (18)
C(14)—C(1)—C(15)	109.17 (22)	105.65 (18)
C(14)—C(1)—C(17)	110.39 (22)	109.14 (18)
C(15)—C(1)—C(17)	107.39 (21)	108.78 (16)
C(1)—C(2)—C(3)	112.47 (23)	112.62 (17)
C(2)—C(3)—C(4)	110.02 (24)	113.26 (17)
C(2)—C(3)—C(12)	110.27 (23)	111.14 (17)
C(4)—C(3)—C(12)	113.47 (22)	109.82 (15)
C(3)—C(4)—C(5)	123.4 (3)	118.02 (18)
C(4)—C(5)—C(6)	124.1 (3)	119.03 (19)
C(5)—C(6)—C(7)	110.59 (23)	113.26 (17)
C(5)—C(6)—C(11)	113.11 (21)	109.79 (17)
C(7)—C(6)—C(11)	111.10 (22)	110.89 (16)
C(6)—C(7)—C(8)	112.74 (22)	114.35 (17)
C(7)—C(8)—C(9)	109.42 (21)	110.68 (17)
C(7)—C(8)—C(19)	110.57 (22)	107.67 (17)
C(7)—C(8)—C(21)	111.44 (22)	111.46 (17)
C(9)—C(8)—C(19)	105.97 (22)	109.28 (17)
C(9)—C(8)—C(21)	110.13 (22)	109.67 (18)
C(19)—C(8)—C(21)	109.17 (20)	107.99 (16)
C(8)—C(9)—C(10)	113.53 (24)	112.22 (18)
C(9)—C(10)—C(11)	112.64 (24)	110.62 (19)
C(6)—C(11)—C(10)	112.57 (21)	108.68 (17)
C(6)—C(11)—C(12)	122.75 (24)	112.62 (16)
C(10)—C(11)—C(12)	124.67 (24)	116.54 (17)
C(3)—C(12)—C(11)	123.13 (23)	112.97 (17)
C(3)—C(12)—C(13)	111.56 (22)	109.67 (15)
C(11)—C(12)—C(13)	125.30 (25)	115.35 (17)
C(12)—C(13)—C(14)	112.72 (24)	110.62 (18)
C(1)—C(14)—C(13)	113.23 (24)	113.62 (19)
C(1)—C(15)—C(3)	111.01 (24)	110.73 (19)
C(1)—C(15)—O(4)	125.9 (3)	125.26 (20)
O(3)—C(15)—O(4)	123.1 (3)	123.92 (21)
C(1)—C(17)—O(1)	110.25 (24)	111.32 (19)
C(1)—C(17)—O(2)	126.1 (2)	124.53 (21)
O(1)—C(17)—O(2)	123.7 (3)	124.15 (20)
C(8)—C(19)—O(5)	111.14 (22)	110.20 (18)
C(8)—C(19)—O(6)	124.6 (3)	125.99 (22)
O(5)—C(19)—O(6)	124.1 (3)	123.71 (20)
C(8)—C(21)—O(7)	110.68 (22)	110.02 (17)
C(8)—C(21)—O(8)	125.65 (25)	126.09 (19)
O(7)—C(21)—O(8)	123.66 (24)	123.88 (19)
C(17)—O(1)—C(18)	116. 5 (3)	115.59 (23)
C(15)—O(3)—C(16)	116.7 (3)	117.38 (24)
C(19)—O(5)—C(20)	117.2 (3)	116.41 (22)
C(21)—O(7)—C(22)	115.35 (24)	117.45 (20)

substituents and the newly formed double bond (Scheme 1). Therefore, the formation of tricyclic (II) with TST stereochemistry is favored. The two structures are relatively symmetric except for the orientation of the ester substituents. In (I), the relative orientations of the two equatorial esters to the tricyclic substructure are slightly different to one another, resulting in the non-symmetric structure. In (II), the axial esters are oriented in opposite directions whereas the equatorial ones are *gauche* to one another. No abnormally short contacts were observed.

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Structure of (±)-Egenine

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Abstract. 6,8-Dihydro-6-(5,6,7,8-tetrahydro-6-methyl-1,3-dioxolo[4,5-g]isoquinolin-5-yl)furo[3,4-e]-1,3-benzodioxol-8-ol, $C_{20}H_{19}NO_6$, $M_r = 369.4$, triclinic, $P\bar{1}$, $a = 10.833$ (2), $b = 13.184$ (3), $c = 13.375$ (3) Å, $\alpha = 89.73$ (3), $\beta = 74.52$ (3), $\gamma = 71.95$ (3)°, $V = 1744.0$ (5) Å^3, $Z = 4$, $D_x = 1.407$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 8.30$ cm^{-1}, $F(000) = 776$, room temperature, $R = 0.051$ for 3375 observed reflections. The relative configuration of the hemiacetal C atom is established. The heterocyclic fragment of isoquinoline exhibits a half-chair conformation; all five-membered rings show envelope conformations. There is one intramolecular hydrogen bond, closing a seven-membered ring.

Introduction. (+)-Egenine, one of the representatives of the phthalideisoquinoline hemiacetals, was isolated from *Fumaria vaillanti* Loisel (Gözler, Gözler & Shamma, 1983). The molecule has three asymmetric centres [C(1), C(14) and C(16)]. The configuration of C(1) and C(14) has been established on chemical grounds (Gözler, Gözler & Shamma, 1983), yet the stereochemistry around the anomeric centre C(16) was not determined.

We have prepared (±)-egenine by Dibal-H reduction of (±)-bicuculline (Rozwadowska & Matecka, 1991). The reduction appeared to be a highly stereospecific process leading exclusively to one of the anomeric hemiacetals. We presumed (Rozwadowska & Matecka, 1991) that the anomeric form which is stabilized by the intramolecular hydrogen bond is formed.

(±)-Egenine
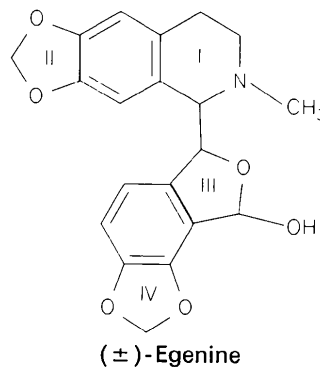
Experimental. Crystals (colourless prisms) were obtained from a methanol and chloroform mixture (1:1). The space group was determined from Weissenberg photographs. A crystal of dimensions 0.1 × 0.15 × 0.2 mm was used for measurements on a Syntex *P*2$_1$ diffractometer, with θ–2θ scans. Cell parameters were determined from least-squares treatment of the setting angles of 15 reflections with $16 \leq 2\theta \leq 27°$. No absorption or extinction corrections were applied. 4622 reflections with $\sin\theta/\lambda \leq 0.55$ Å^{-1} were measured in the range $h\,0\rightarrow14$, $k\,-11\rightarrow11$, $l\,-14\rightarrow14$. Two standard reflections ($\bar{3}12$ and $3\bar{1}1$), recorded every 98 measurements, showed intensity variation of ± 6%. 3375 reflections were observed with $I \geq 4\sigma(I)$; peak profile analysis was according to