

Table 6. Torsion angles (°) describing the conformation of the molecule

Theophylline		Pyranose ring		Others	
N(1)—C(6)—C(5)—C(4)	3.0 (3)	O(0')—C(1')—C(2')—C(3')	11.1 (2)	H(3')—C(3')—C(4')—H(4')	−43.2 (1)
C(2)—N(1)—C(6)—C(5)	−4.0 (3)	C(1')—C(2')—C(3')—C(4')	6.6 (2)	C(3')—C(4')—C(5')—C(6')	−167.5 (1)
N(3)—C(2)—N(1)—C(6)	2.6 (3)	C(2')—C(3')—C(4')—C(5')	11.4 (2)	C(3')—C(4')—C(5')—H(5')	72.7 (1)
C(4)—N(3)—C(2)—N(1)	0.0 (3)	C(3')—C(4')—C(5')—O(0')	−47.0 (2)	N(7)—C(4')—C(5')—O(0')	77.1 (2)
C(5)—C(4)—N(3)—C(2)	−1.0 (3)	C(4')—C(5')—O(0')—C(1')	68.4 (2)	H(4')—C(4')—C(5')—H(5')	−46.2 (1)
C(6)—C(5)—C(4)—N(3)	0.0 (3)	C(5')—O(0')—C(1')—C(2')	−48.8 (2)	O(0')—C(5')—C(6')—O(3')	82.5 (2)
C(4)—C(5)—N(7)—C(8)	0.0 (2)			C(5')—C(6')—O(3')—C(10')	−147.3 (2)
C(5)—N(7)—C(8)—N(9)	0.0 (2)			C(6')—O(3')—C(10')—C(11')	−170.8 (2)
N(7)—C(8)—N(9)—C(4)	0.0 (2)	O(0')—C(1')—C(2')—N(1')	−169.2 (1)	C(6')—O(3')—C(10')—O(4')	7.8 (3)
C(8)—N(9)—C(4)—C(5)	0.0 (2)	O(1')—C(1')—C(2')—C(3')	−111.9 (2)	O(0')—C(1')—O(1')—C(7')	67.1 (2)
N(9)—C(4)—C(5)—N(7)	0.0 (2)	H(1')—C(1')—C(2')—C(3')	127.1 (2)	C(1')—C(2')—N(1')—C(8')	−170.9 (2)
		C(1')—C(2')—C(3')—H(3')	−180.0 (2)	C(2')—N(1')—C(8')—O(2')	−8.6 (3)
		C(2')—C(3')—C(4')—N(7)	−113.1 (2)	C(2')—N(1')—C(8')—C(9')	170.4 (2)
		C(2')—C(3')—C(4')—H(4')	130.4 (1)	C(8)—N(7)—C(4')—C(5')	−81.0 (2)

USA, for the facilities placed at her disposal, and Dr N. Pravdić for the crystal and valuable discussions.

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The Crystal and Molecular Structure of 7-(Methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy- β -D-erythro-hex-2-enopyranosid-4-yl)theophylline, C₁₈H₂₃N₅O₇

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Abstract

The title compound crystallizes in the monoclinic space group *C*2 with $a = 24.093(1)$, $b = 5.003(3)$, $c = 19.002(2)$ Å, $\beta = 118.85(4)^\circ$, $Z = 4$. The structure was refined to an R of 0.051. The orientation of the base relative to the sugar ring, defined in terms of rotation about the C(4')—N(7) glycosyl bond, is *syn*

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(105.1°). The theophylline base is planar within the limits of experimental error. The sugar moiety exhibits a half-chair 5H_0 conformation. The molecules are connected by N(1')—H...O(2), 3.09 Å, hydrogen bonds acting between the sugar and base moieties forming an infinite chain along **a**. Base stacking occurs along **b**.

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Experimental

The space group was determined from Weissenberg photographs recorded with Cu $K\alpha$ radiation. The diffraction symmetry and space-group extinctions indicated $C2$ or Cm ; the structure was solved and refined successfully in $C2$. Table 1 lists the crystallographic and physical data.

The intensities were collected on a Philips PW 1100 computer-controlled four-circle diffractometer in the ω -scan mode [scan width = 1.20° (θ), scan speed = 0.03° (θ) s^{-1}] with graphite-monochromated Cu $K\alpha$ radiation. 1270 independent reflexions [$I > 2\sigma(I)$] in the range $2 < \theta < 70^\circ$ were used in the calculations. Three standard reflexions were measured every 2 h. The data were corrected for background, Lorentz and polarization effects but not for absorption.

Structure determination and refinement

An overall temperature factor ($B = 2.04 \text{ \AA}^2$) and a scale factor were determined (Wilson, 1942) and used to compute normalized structure amplitudes by the routine *NORMAL* included in *MULTAN*. The geometry of the planar molecular fragment (11 atoms of the theophylline base) was used to modify the Wilson plot by applying the spherically averaged molecular scattering factor. 300 reflexions with $|E| > 1.15$ were used. *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) failed; no set of phases yielded an E map in which all or even a recognizable fragment appeared. Particular tactics described by Lessinger (1976) were applied but without success. The *MULTAN* failure could be attributed to the relatively small number of recorded intensities and difficulties typical of the $C2$ space group. To find a solution a magic-integer representation for a symbolic phase assignment with the P,S method (Woolfson, 1976; Main, 1977) was employed to derive phases. The values for 42 phases were obtained and put as known phases into *MULTAN* (Main *et al.*, 1977). 4000 \sum_2 phase relationships were used. The relative weights for ABS FOM, PSI ZERO and RESID were used; ABS FOM

was given less and PSI ZERO more weight. The set with ABS FOM = 1.304, PSI ZERO = 33.2, RESID = 31.44 was selected and revealed 29 non-hydrogen atoms out of 30 in the molecule. The same fragment occurred in the E map using 15 known phases with $|E| > 2.0$ in the *MULTAN* input. The remaining C atom belonging to the terminal methyl group was located from the resulting Fourier synthesis.

Refinement was by a full-matrix least-squares method minimizing $\sum w|F_o| - |F_c|^2$ with $w = 1/\sigma^2_{F_o}$. Anisotropic refinement and a subsequent difference synthesis were performed to locate all H atoms. In the final cycles one scale factor, and the positional and anisotropic thermal parameters (269 in all) were varied. The H atoms were included only in the structure factor calculations. For the H atoms, the isotropic thermal parameters were those of the bonded atom. The final $R = 0.051$ and $R_w = 0.064$. The analysis of the title compound is of limited accuracy because of the poor quality of the crystal.

Scattering factors given by Cromer & Mann (1968) and (for H) Stewart, Davidson & Simpson (1965) were used.

The calculations were performed on a Univac 1110 computer at the University Computing Centre in Zagreb with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

Table 2. Final atomic parameters ($\times 10^4$) for the non-hydrogen atoms

	x	y	z
C(1)	2556 (3)	-1777 (23)	3120 (5)
C(2)	1803 (3)	-4451 (16)	1969 (4)
C(3)	1054 (3)	-6985 (19)	780 (4)
C(4)	702 (3)	-3822 (16)	1472 (4)
C(5)	805 (3)	-1912 (16)	2023 (4)
C(6)	1420 (3)	-1075 (0)	2599 (4)
C(8)	-212 (3)	-2523 (16)	1231 (4)
C(1')	-924 (3)	-1160 (15)	2657 (4)
C(2')	-1063 (3)	953 (16)	2019 (4)
C(3')	-627 (3)	1801 (16)	1828 (4)
C(4')	48 (3)	915 (16)	2325 (4)
C(5')	186 (3)	-215 (15)	3127 (4)
C(6')	245 (3)	1971 (16)	3729 (4)
C(7')	-1054 (4)	-1963 (23)	3786 (5)
C(8')	-1967 (3)	3639 (16)	1039 (4)
C(9')	-2657 (3)	4170 (20)	747 (4)
C(10')	1351 (3)	3016 (16)	4459 (4)
C(11')	1845 (3)	4965 (18)	4531 (5)
N(1)	1895 (2)	-2500 (15)	2530 (3)
N(3)	1191 (2)	-5083 (14)	1426 (3)
N(7)	205 (2)	-1044 (12)	1869 (3)
N(9)	74 (2)	-4277 (15)	975 (3)
N(1')	-1708 (2)	1688 (13)	1625 (3)
O(2)	2246 (2)	-5596 (12)	1949 (3)
O(6)	1557 (2)	720 (0)	3099 (3)
O(0')	-300 (2)	-2104 (10)	3014 (2)
O(1')	-1071 (2)	26 (12)	3228 (3)
O(2')	-1674 (2)	4803 (14)	771 (3)
O(3')	776 (2)	3679 (11)	3866 (3)
O(4')	1444 (2)	1067 (14)	4878 (3)

Table 1. Crystallographic and physical data

Numbers in parentheses here and throughout this paper are the e.s.d.'s in the least significant digits.

7-(Methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy- β -D-erythro-hex-2-enopyranosid-4-yl) theophylline, $C_{18}H_{23}N_5O_7$

FW	437.42	U	$2006 \cdot 17 \text{ \AA}^3$
Space group	$C2$	Z	4
a	24.093 (1) \AA	D_c	1.407 Mg m^{-3}
b	5.003 (3)	$\mu(\text{Cu } K\alpha)$	1.137 mm^{-1}
c	19.002 (2)	Crystal shape	Needle
β	118.85 (4) $^\circ$	Crystal size	$0.028 \times 0.042 \times 0.566 \text{ mm}$

Atomic positional parameters are listed in Tables 2 and 3.*

Description and discussion of the structure

The present structure determination is part of an investigation of the new type of 2',3'-unsaturated nucleosides with the base linked at C(4') (Kojić-Prodić, Danilov & Pravdić, 1976; Kojić-Prodić, 1979; Pravdić, 1979).

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34188 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Positional parameters ($\times 10^3$) for the hydrogen atoms

	x	y	z
H(1)1	258	000	350
H(1)2	267	-350	325
H(1)3	267	-70	275
H(3)1	117	-900	100
H(3)2	133	-733	67
H(3)3	58	-767	50
H(8)	-65	-233	92
H(1')	-125	-280	242
H(3')	-81	300	125
H(4')	33	260	242
H(5')	58	-167	342
H(6')1	-15	333	350
H(6')2	42	100	433
H(7')1	-137	-367	367
H(7')2	-56	-267	417
H(7')3	-100	-40	425
H(9')1	-281	247	100
H(9')2	-281	500	25
H(9')3	-268	533	120
H(11')1	195	367	413
H(11')2	217	533	510
H(11')3	167	667	425
H(N1')	-212	100	170

Fig. 1. The structural formula and intramolecular distances (Å).

The structural formula with the atom numbering and interatomic distances is shown in Fig. 1. Bond angles are listed in Table 4. The molecular packing and hydrogen bonds are illustrated in Fig. 2. The conformation of the carbohydrate moiety is shown in Fig. 3 and Tables 5 and 6. Torsion angles defining the conformation of the molecule are presented in Table 6.

Table 4. Bond angles ($^\circ$)

C(1)-N(1)-C(2)	118.0 (6)	C(3')-C(4')-C(5')	112.9 (6)
C(1)-N(1)-C(6)	115.5 (6)	C(3')-C(4')-N(7)	109.6 (5)
C(2)-N(1)-C(6)	126.5 (4)	C(5')-C(4')-N(7)	110.7 (6)
N(1)-C(2)-O(2)	122.3 (5)	O(0')-C(5')-C(4')	109.3 (4)
N(1)-C(2)-N(3)	117.3 (6)	O(0')-C(5')-C(6')	110.9 (6)
O(2)-C(2)-N(3)	120.4 (7)	C(4')-C(5')-C(6')	112.4 (6)
C(2)-N(3)-C(3)	120.6 (6)	O(3')-C(6')-C(5')	108.1 (6)
C(2)-N(3)-C(4)	119.4 (6)	N(1')-C(8')-O(2')	123.7 (6)
C(3)-N(3)-C(4)	120.0 (5)	N(1')-C(8')-C(9')	114.3 (7)
N(3)-C(4)-C(5)	122.2 (5)	O(2')-C(8')-C(9')	122.0 (7)
N(3)-C(4)-N(9)	125.3 (6)	O(3')-C(10')-O(4')	122.7 (7)
C(5)-C(4)-N(9)	112.5 (6)	O(3')-C(10')-C(11')	111.7 (6)
C(4)-C(5)-N(7)	105.9 (5)	O(4')-C(10')-C(11')	125.5 (5)
C(4)-C(5)-C(6)	122.7 (6)	C(1')-O(1')-C(7')	110.2 (7)
C(6)-C(5)-N(7)	131.4 (7)	C(6')-O(3')-C(10')	118.0 (6)
N(1)-C(6)-C(5)	112.0 (5)	C(2')-N(1')-C(8')	124.4 (6)
N(1)-C(6)-O(6)	120.9 (5)	N(7)-C(8)-H(8)	129
C(5)-C(6)-O(6)	127.1 (6)	N(9)-C(8)-H(8)	117
C(5)-N(7)-C(8)	105.2 (6)	C(2')-N(1')-H(N1')	132
C(5)-N(7)-C(4')	128.0 (5)	C(8')-N(1')-H(N1')	104
N(8)-N(7)-C(4')	126.6 (5)	O(0')-C(1')-H(1')	110
N(7)-C(8)-N(9)	112.8 (5)	O(1')-C(1')-H(1')	104
C(4)-N(9)-C(8)	103.3 (6)	C(2')-C(1')-H(1')	111
C(1')-O(0')-C(5')	117.2 (5)	C(2')-C(3')-H(3')	116
O(0')-C(1')-O(1')	112.0 (4)	C(4')-C(3')-H(3')	124
O(0')-C(1')-C(2')	113.1 (6)	C(3')-C(4')-H(4')	107
O(1')-C(1')-C(2')	106.2 (6)	C(5')-C(4')-H(4')	109
N(1')-C(2')-C(1')	111.6 (6)	N(7)-C(4')-H(4')	108
N(1')-C(2')-C(3')	125.7 (6)	O(0')-C(5')-H(5')	95
C(1')-C(2')-C(3')	122.5 (6)	C(4')-C(5')-H(5')	118
C(2')-C(3')-C(4')	119.5 (6)	C(6')-C(5')-H(5')	110

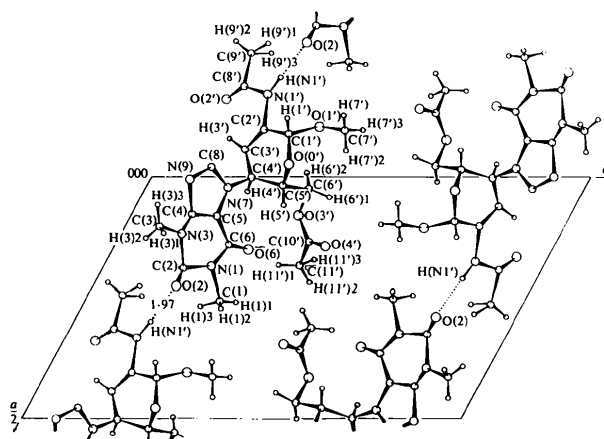


Fig. 2. Molecular packing viewed along *b*. Intermolecular hydrogen bonds between the amide H atom (in the sugar moiety) and the keto O atom (in the base moiety) are represented by dotted lines. The N(1')...O(2') distance is 3.09 Å. A right-handed coordinate system has been chosen so that the positive direction of *b* is away from the reader.

In the preparation of the title compound, the *D* compound was used as the starting material. In the present structure determination the *D* enantiomer was selected and the configuration and conformation were defined in accordance with its torsion angles (Table 6).

Base stacking is shown in Fig. 4.

Base

The theophylline base (1,3-dimethylxanthine) realizes the glycosyl bond through the *N*(7)H form. The e.s.d.'s of bond distances and angles are much

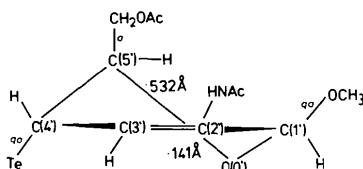


Fig. 3. Schematic drawing of the ⁵H₀ half-chair conformation illustrating the displacements of C(5') (above) and O(0') (below) the plane defined by C(1'), C(2'), C(3') and C(4'), and the orientation of the substituents.

Table 5. Displacements from least-squares planes through the base and sugar (Å)

Atoms included in the calculation of the planes are denoted by an asterisk.

Theophylline		Sugar	
N(1)*	0.005 (11)	C(1')*	-0.016 (11)
C(2)*	-0.003 (13)	C(2')*	0.034 (11)
N(3)*	-0.015 (11)	C(3')*	-0.034 (11)
C(4)*	0.012 (12)	C(4')*	0.015 (11)
C(5)*	0.012 (12)	C(5')	0.532 (11)
C(6)*	-0.005 (14)	O(0')	-0.141 (7)
N(7)*	-0.005 (1)	N(1')	0.044 (9)
C(8)*	-0.014 (13)		
N(9)*	0.013 (11)		
O(2)	0.003 (10)		
O(6)	-0.063 (5)		
C(1)	0.010 (17)		
C(3)	-0.118 (15)		

larger than those for the α -*D-threo* isomer (Kojić-Prodć, 1979).

The base conformation is defined by the mean torsion angles of 1 and 0.8° for the six- and five-membered rings, respectively (Table 6). The base is almost planar; the displacements of the atoms from the least-squares planes (Table 5) support this fact.

Sugar moiety

Bond lengths and angles (Fig. 1 and Table 4) are comparable with the values found in the α -*D-threo* isomer (Kojić-Prodć, 1979) and also in some peracetylated 2,3-dideoxyaldopyranoses (Rogić, 1975; Kojić-Prodć, Rogić & Ružić-Toroš, 1976). Lower accuracy of the data prevents us from drawing any conclusion on the C—O distances associated with the anomeric C atom. The endocyclic C(5')—O(0')—C(1') bond angle of 117.2° is rather large although a value of 116.1° was found in sucrose (α -*D-glucose* residue) (Hybl, Rundle & Williams, 1965).

The carbohydrate moiety exhibits the ⁵H₀ half-chair conformation (Fig. 3), possible for β -anomers (Stoddart, 1971). The best least-squares plane is defined by C(1'), C(2'), C(3') and C(4'); C(5') and O(0') are displaced from this plane by 0.532 and -0.141 Å, respectively (Table 5). The ring substituents are attached at C(1') and C(4') in quasi-axial and C(5') in

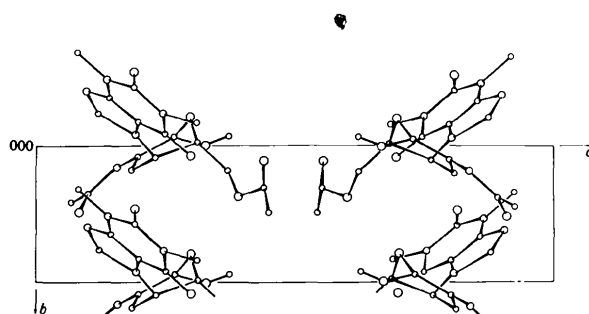


Fig. 4. Base stacking along **b** with the shortest interbase distance of 5.003 Å.

Table 6. Torsion angles (°) describing the conformation of the molecule

Theophylline		Pyranose ring			
N(1)—C(6)—C(5)—C(4)	-1 (1)	O(0')—C(1')—C(2')—C(3')	0 (1)	H(3')—C(3')—C(4')—H(4')	50
C(2)—N(1)—C(6)—C(5)	2 (1)	C(1')—C(2')—C(3')—C(4')	-8 (1)	C(3')—C(4')—C(5')—C(6')	-75.8 (8)
N(3)—C(2)—N(1)—C(6)	0 (1)	C(2')—C(3')—C(4')—C(5')	-17 (1)	C(3')—C(4')—C(5')—H(5')	154
C(4)—N(3)—C(2)—N(1)	-2 (1)	C(3')—C(4')—C(5')—O(0')	47.7 (8)	N(7)—C(4')—C(5')—O(0')	-75.6 (7)
C(5)—C(4)—N(3)—C(2)	2 (1)	C(4')—C(5')—O(0')—C(1')	-59.3 (8)	H(4')—C(4')—C(5')—H(5')	-87
C(6)—C(5)—C(4)—N(3)	-1 (1)	C(5')—O(0')—C(1')—C(2')	35.3 (8)	O(0')—C(5')—C(6')—O(3')	175.3 (5)
C(4)—C(5)—N(7)—C(8)	-0.1 (8)			C(5')—C(6')—O(3')—C(10')	-89.4 (7)
C(5)—N(7)—C(8)—N(9)	0.8 (8)		Others	C(6')—O(3')—C(10')—C(11')	-179.5 (7)
N(7)—C(8)—N(9)—C(4)	-1.2 (8)	O(0')—C(1')—C(2')—N(1')	174.9 (6)	C(6')—O(3')—C(10')—O(4')	0 (1)
C(8)—N(9)—C(4)—C(5)	1.1 (9)	O(1')—C(1')—C(2')—C(3')	123.0 (8)	O(1')—C(1')—O(1')—C(7')	-65.6 (7)
N(9)—C(4)—C(5)—N(7)	-0.7 (9)	H(1')—C(1')—C(2')—C(3')	-125	C(1')—C(2')—N(1')—C(8')	177.4 (7)
		C(1')—C(2')—C(3')—H(3')	166	C(2')—N(1')—C(8')—O(2')	4 (1)
		C(2')—C(3')—C(4')—N(7)	107.2 (8)	C(2')—N(1')—C(8')—C(9')	-176.5 (7)
		C(2')—C(3')—C(4')—H(4')	-136	C(8)—N(7)—C(4')—C(5')	105.1 (7)

axial positions (Fig. 3) (Stoddart, 1971), as expected for the ⁵H₀ half-chair conformation. The highly distorted conformation appears to be due to the departure of C(5') from the plane to avoid steric hindrance between bulky substituents at the contiguous C(4') and C(5').

The puckering of the sugar ring required by the ⁵H₀ conformation can be described by the values of the C(2')—C(3')—C(4')—C(5') (−17°) and C(3')—C(4')—C(5')—O(0') (47.7°) torsion angles (Table 6).

Sundaralingam (1968) has defined the conformation about C(5)—C(6) in pyranosides by the angle φ₀₀ = O(5)—C(5)—C(6)—O(6). In the present structure this angle is described by the sequence O(0')—C(5')—C(6')—O(3') and its value of 175.3° is not in the range common to pyranoside derivatives (±60 ± 30°).

Nucleoside conformation and molecular packing

The orientation of the base relative to the sugar ring described in terms of rotation about the N(7)—C(4') glycosyl bond for the sequence C(8)—N(7)—C(4')—C(5') is *syn* (105.1°) (Sundaralingam, 1975). The conformation about the glycosyl bond with the torsion angle (χ) larger than 90° can also be referred to as *high-anti* (90–130°) (Sundaralingam, 1975). Thus, a comparison of the molecules in the usual *anti* range can be made.

The molecules are joined by N(1')—H...O(2) (3.09 Å) hydrogen bonds acting between the sugar and base moieties (Fig. 2). Each molecule is connected by two hydrogen bonds with the neighbouring molecule forming infinite chains along **a**.

Base stacking with the shortest interbase distance of 5.003 Å occurs along **b**.

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The Crystal Structures of Two Fluorene Derivatives

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Abstract

The crystal structures of 2,7-dibromo-9-[4-(dimethylamino)benzylidene]fluorene (I) (C₂₂H₁₇Br₂N) and 9,9'-(1,4-phenylenedimethyldiyl)difluorene (II) (C₃₄H₂₂) were determined by X-ray structure analysis. The

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crystals are monoclinic, space group *P2₁/c*, with *a* = 8.200 (6), *b* = 14.536 (7), *c* = 15.729 (5) Å, β = 103.64 (6)° and *Z* = 4 for (I), and orthorhombic, space group *Pbca*, with *a* = 17.640 (6), *b* = 23.630 (6), *c* = 10.755 (5) Å and *Z* = 8 for (II). The structures were solved by Patterson methods for (I) and direct

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