Chapter 5

DETERMINATION OF THE STRUCTURE

The tungsten vanadium oxide is composed of tungsten, vanadium and oxygen which atomic numbers are 74, 23 and 8 respectively. Since the atomic number of tungsten is much larger than those of vanadium and oxygen, the structure of tungsten vanadium oxide can be determined by heavy atom method.

Numerical computations were made on IBM 3031-008 system (OS/VSI) using standard crystallographic program.[32] All programs were written in FORTRAN IV. The details of the programs used are listed in Table 5.1 Computations with these programs were carried out at the Computer Service Centre, Chulalongkorn University.

Table 5.1 Programs used in performing the crystallographic calculations on IBM 3031-008 system (OS/VSI)

Program	Calculation	Authors
ABSW	Lorentz-polarization and absorption corrections on X-ray data collected in a two-circle (Weissenberg) geometry crystals can be of shperical,cylindrical or general shape.	J-O LUNDGREN
UPALS	Calculation of structure factors and full-matrix least-square refinements.	Idem.

Table 5.1 (continued)

Program	Calculation	Authors
FORDUP	Fourier syntheses maps can be calculated in planes parallel to unit cell axes or in planes defined by three non-colinear points, arbitrarily chosen in the unit cell. F_o,F_c, difference maps : reflexion input is from an UPALS output file. Patterson maps : reflection input is from a standard file or an UPALS output file.	Idem.
DISTAN	Calculation of distances and angles with E.S.D.'s. An input file for this program may be writtenin UPALS.	Idem.

Intensity Data Reduction

From Weissenberg intensity data of 238 reflections of hol, $\bar{h}$ol and 231 reflections of h21, $\bar{h}$21, Lorentz and polarization correction as well as absorption correction using the Gaussian grid method applied to obtain the square of observed structure factor amplitudes by using the ABSW program. The linear absorption coefficient of $W_3V_5O_{20}^{\circ}$ for MoK_α- radiation is 287 cm^{-1} which was derived from the Eq. 5.1 as follows :

$$\mu_\lambda = \rho \sum_n^{P_n} (\frac{P_n}{100}) (\frac{\mu}{\rho})_{\lambda,E_n} \quad \text{..... 5.1}$$

where μ_λ is the linear absorption, (μ/ρ) is defined as the mass absorption coefficient for the wavelength used and taken from the International Table, Vol.III, P. 162 and $\frac{P_n}{100}$ is number of percent of an element of a compound. The transmission factors varied between 0.2576 and 0.3696

All of the 469 reflections were used for the calculation of Patterson function, F_o synthesis and least square refinements.

Determination of Heavy Atom Positions by Using Patterson Function

It is shown from experimental data (chapter 4) that $W_3V_5O_{20}$ crystallizes in monoclinic system. A unit cell consists of six atoms of tungsten, ten vanadium atoms and forty oxygen atoms. The space group of $W_3V_5O_{20}$ crystals may be one of three space groups (C2,Cm,C2/m).

By considering unit cell parameters obtained from oscillation, Weissenberg and precession photographs, it is found that these values are close to the unit cell parameters of WV_2O_7 by Mondet[3] et al. and of $W_3V_5O_{20}$ by Kihlborg[4] et al. (Table 5.2)

Table 5.2 Comparing unit cell parameters from this work with the values by Mondet et al. and the values by Kihlborg et al.

unit cell parameters	By Mondet et al.	By Kihlborg et al.	From this work
a (Å)	24.4	24.413 (3)	24.412 (2)
b (Å)	7.44	7.446 (2)	7.4479 (8)
c (Å)	3.95	3.950 (1)	3.9506 (3)
β (degree)	90	91.03 (2)	91.027 (7)

In order to find out the most probable space group and the structure model for this V-W-O compound, the calculation of structure factors of these two models using UPALS X-ray data from this work were made. These yielded an R value of 0.398 for $W_3V_5O_{20}$ model lower than that of 0.447 for WV_2O_7 model. Since the space group of WV_2O_7 is C2 and the space group of $W_3V_5O_{20}$ is C2/m, C2 should not be the space group of the model in this work. Only two space groups, C2/m and Cm, therefore, would be used for $W_3V_5O_{20}$ in determining heavy atomic positions.

The atomic positions of $W_3V_5O_{20}$ were determined from the Patterson synthesis. It is seen from the interatomic vectors derived from general positions of C2/m and Cm as shown in Table 5.3 and Table 5.4 that the Harker planes (uow) and (u ½ w) occured only in space group C2/m whereas the Harker lines (ovo) and (½ vo) appears both in space groups C2/m and Cm. So it is not possible to find the coordinates x and z from the Patterson sections with the space group Cm.

The Patterson section P (uow), (u ½ w) and (uvo) were evaluated by the FORDUP program using all of 469 reflections. The calculations were performed at sections uow, u 0.5 w, with the fractional grid intervals of 0.02 along "u" and "w" and at the section uvo with the fractional grid intervals of 0.02 along "u" and "v". These maps are shown in Fig. 5.1

According to the program, the height of the origin peak is normalized to 999, the expected peak height for the various Patterson maxima can be approximately determined. The Patterson's peak height maxima for a pair of atoms proportional to the products of the atomic numbers of the atoms they join. The approximated peak height maxima

Table 5.3 Interatomic vectors derived from a general equivalent position 8j in the space group C2/m

	x,y,z	x,-y,z	-x,y,-z	-x,-y-z	½+x,½+y,z	½+x,½-y,z	½-x,½+y,-z	½-x,½-y,-z
x,y,z	0,0,0	0,-2y,0	-2x,0,-2z	-2x,-2y,-2z	½,½,0	½,½-2y,0	½-2x,½,-2z	½-2x,½-2y,-2z
x,-y,z	0,2y,0	0,0,0	-2x,2y,-2z	-2x,0,-2z	½,½+2y,0	½,½,0	½-2x,½+2y,-2z	½-2x,½,-2z
-x,y,-z	2x,0,2z	2x,-2y,2z	0,0,0	0,-2y,0	½+2x,½,2z	½+2x,½-2y,2z	½,½,0	½,½-2y,0
-x,-y,-z	2x,2y,2z	2x 0 2z	0 2y 0	0,0,0	½+2x,½+2y,2z	½+2x,½,2z	½,½+2y,0	½,½,0
½+x,½+y,z	-½,-½,0	-½,-½,0	-½-2x,-½,-2z	-½-2x,-½-2y,-2z	0,0,0	0, -2y, 0	-2x,0,-2z	-2x,-2y,-2z
½+x,½-y,z	-½,-½+2y,0	-½,-½,0	-½-2x,-½,-2z	-½-2x,-½,-2z	0,2y,0	0,0,0	-2x,-2y,-2z	-2x,0,-2z
½-x,½+y,-z	-½+2x,-½,2z	-½+2x,-2y,2z	-½,-½,0	-½,-½-2y,0	2x,0,2z	2x,2y,2z	0,0,0	0,-2y 0
½-x,½-y,z	-½+2x,½+2y,2z	-½+2x,-½+2y,2z	-½,-½+2y,0	-½,-½,0	2x,2y,2z	2x 0 2z	0,-2y,0	0,0,0

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Table 5.4 Interatomic vectors derived from a general equivalent positions 4b in the space group Cm

	x,y,z	x,-y,z	½+x,½+y,z	½+x,½-y,z
x,y,z	0,0,0	0,2y,0	½,½,0	½,½-2y,0
x,-y,z	0,2y,0	0,0,0	½,½+2y,0	½,½,0
½+x,½+y,z	-½,-½,0	-½,-½,0	0,0,0	0,-2y,0
½+x,½-y,z	-½,-½+2y,0	-½,-½,0	0,2y,0	0,0,0

in the Patterson map between two atoms are listed in Table 5.5

Table 5.5 The approximated peak height maxima in Patterson map of $W_3V_5O_{20}$

Type to atoms	The approximated height maxima
origin peak	999
W-W	134
W-V	42
V-V	13
W-O	15
V-O	5
O-O	2

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From the Patterson maps, there was only one peak that should be correspond to tungsten-tungsten peak. This was the peak of height 373 on the Harker plane (u,o,w) and the other peak of height 999 on the Harker line (½ vo). These peaks should correspond to tungsten - tungsten peak. Thus the following assignments could be drawn :

a. Section (u,o,w),

peak found at 0.14 0 0.16

assigned $\pm$ 2x, 0, $\pm$ 2z SO x = 0.07, z = 0.08 and x = 0.93, z = 0.92

b. Section (u,v,o),

peak found at 0, 0.5, 0 and

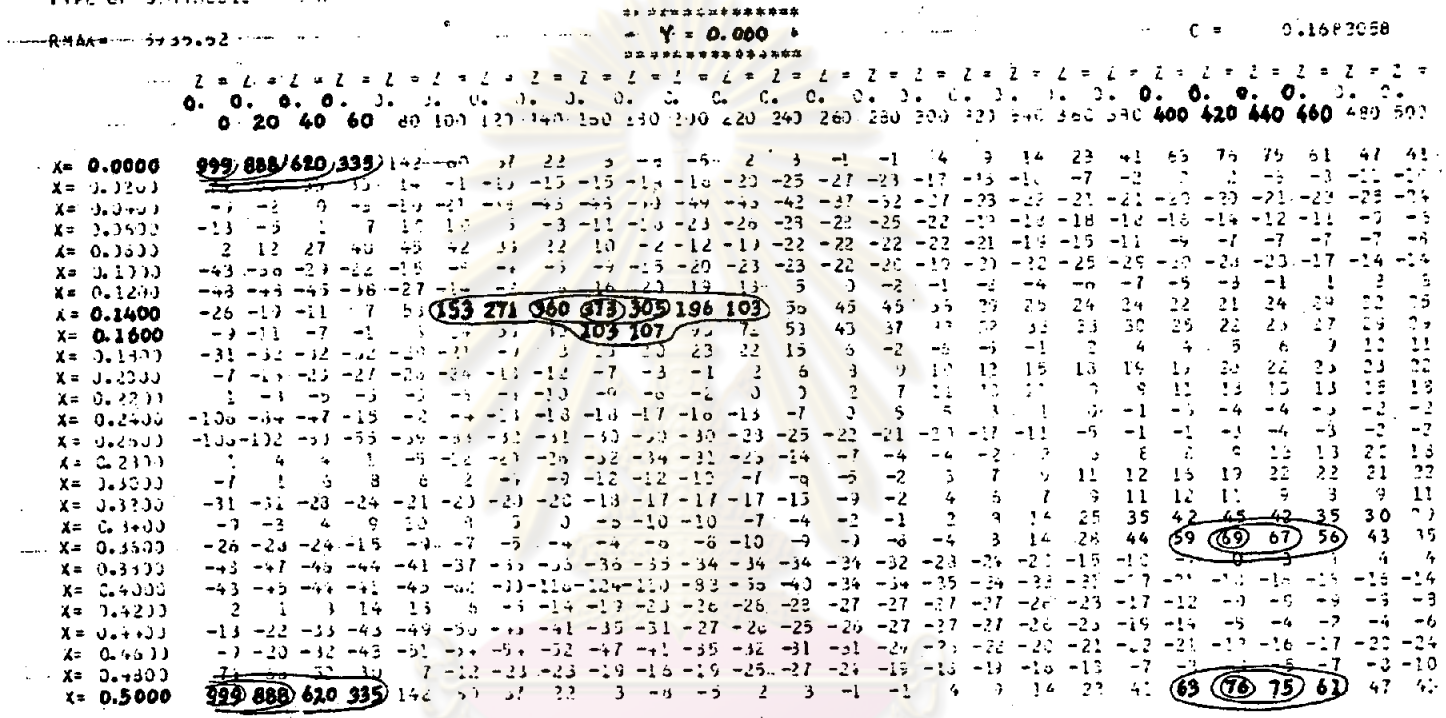
assigned 0, $\pm$ 2y, 0 and $\pm$ ½, $\pm$ (½-2y), 0

hence y = 0.25 or 0.75

The coordinates of atoms obtained from the above assignments are 0.07, 0.25, 0.08; 0.07, 0.75, 0.08; 0.93, 0.25, 0.92 and 0.93, 0.75, 0.92. The last three coordinates are equivalent positions with the first by symmetry. The coordinates of 8 tungsten atoms W1 obtained from Patterson maps, therefore should be at 0.07, 0.25, 0.08 in 8j position of space group C2/m or assumed 4 tungsten atoms at 0.07, 0.25, 0.08 in position 4b of space group Cm.

Determination of Other Atoms by the Fo Synthesis

Fourier calculations using the observed structure factor amplitudes with the calculated phases as the coefficients are called the Fo synthesis. This can be expressed in the form



(a)

Fig. 5.1 Patterson maps of $W_3V_5O_{20}$

(a) P (uow)

(b) P (uvo)

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VANADIUM TUNGSTATE V5W3O20 CELL FORDAP
TYPE OF SYNTHESIS = PATT

RMAX= 3935.65 ********** * Z = 0.000 * ********** C = 0.15332052

	Y = 0. 0	Y = 0. 20	Y = 0. 40	Y = 0. 60	Y = 0. 80	Y = 0. 100	Y = 0. 120	Y = 0. 140	Y = 0. 160	Y = 0. 180	Y = 0. 200	Y = 0. 220	Y = 0. 240	Y = 0. 260	Y = 0. 280	Y = 0. 300	Y = 0. 320	Y = 0. 340	Y = 0. 360	Y = 0. 380	Y = 0. 400	Y = 0. 420	Y = 0. 440	Y = 0. 460	Y = 0. 480	Y = 0. 500
X= 0.0000	999	979	921	828	707	564	403	251	101	-33	-141	-217	-256	-256	-217	-141	-33	101	251	408	563	706	828	921	979	999
X= 0.0200	74	72	69	62	54	45	35	25	15	6	-1	-6	-9	-9	-6	-1	6	15	25	35	45	55	62	69	72	74
X= 0.0400	-9	-9	-9	-9	-8	-8	-7	-6	-6	-5	-5	-4	-4	-4	-4	-5	-5	-6	-6	-7	-8	-8	-9	-9	-9	-9
X= 0.0600	-13	-13	-13	-13	-13	-13	-13	-13	-13	-13	-12	-12	-12	-12	-12	-12	-12	-12	-12	-12	-12	-12	-13	-13	-13	-13
X= 0.0800	2	1	0	-2	-5	-8	-11	-14	-17	-20	-23	-24	-25	-25	-24	-22	-20	-17	-14	-11	-8	-5	-2	0	1	2
X= 0.1000	-43	-42	-40	-36	-35	-31	-26	-22	-18	-14	-11	-9	-8	-8	-9	-11	-14	-18	-22	-26	-31	-34	-38	-40	-42	-43
X= 0.1200	-43	-43	-42	-47	-46	-45	-44	-43	-42	-41	-41	-40	-40	-40	-40	-41	-41	-42	-43	-44	-45	-46	-47	-47	-43	-48
X= 0.1400	-26	-27	-28	-30	-35	-37	-41	-45	-49	-52	-55	-57	-58	-58	-57	-55	-52	-49	-45	-41	-37	-34	-30	-28	-26	-26
X= 0.1600	-9	-9	-9	-10	-10	-10	-10	-11	-11	-11	-11	-11	-12	-12	-11	-11	-11	-11	-11	-10	-10	-10	-10	-9	-9	-9
X= 0.1800	-31	-31	-29	-26	-22	-17	-12	-7	-2	2	6	8	10	10	6	6	2	-2	-7	-12	-17	-22	-26	-29	-31	-31
X= 0.2000	-7	-7	-8	-9	-10	-12	-13	-15	-16	-18	-19	-19	-20	-20	-19	-19	-17	-16	-15	-13	-12	-10	-9	-8	-7	-7
X= 0.2200	1	1	2	4	6	9	12	15	18	21	23	24	25	25	24	23	21	18	15	12	9	6	4	2	1	1
X= 0.2400	-106	-98	-78	-34	17	78	143	209	273	329	374	407	423	423	407	375	329	273	210	143	78	18	-34	-78	-98	-106
X= 0.2600	-106	-98	-78	-34	17	78	143	209	273	329	375	407	423	423	407	375	329	273	210	143	78	18	-34	-78	-97	-106
X= 0.2800	1	1	2	4	6	9	12	15	18	21	23	24	25	25	24	23	21	18	15	12	9	6	4	2	1	1
X= 0.3000	-7	-7	-8	-9	-10	-12	-13	-15	-16	-18	-19	-19	-20	-20	-19	-19	-17	-16	-15	-13	-12	-10	-9	-8	-7	-7
X= 0.3200	-31	-31	-29	-26	-22	-17	-12	-7	-2	2	6	8	10	10	6	6	2	-2	-7	-12	-17	-22	-26	-29	-31	-31
X= 0.3400	-9	-9	-9	-10	-10	-10	-10	-11	-11	-11	-11	-11	-12	-12	-11	-11	-11	-11	-11	-10	-10	-10	-10	-9	-9	-9
X= 0.3600	-26	-27	-28	-30	-33	-37	-41	-45	-49	-52	-55	-57	-58	-58	-57	-55	-52	-49	-45	-41	-37	-33	-30	-28	-26	-26
X= 0.3800	-43	-43	-42	-47	-46	-45	-44	-43	-42	-41	-41	-40	-40	-40	-40	-41	-41	-42	-43	-44	-45	-46	-47	-47	-48	-49
X= 0.4000	-43	-42	-40	-38	-35	-31	-26	-22	-18	-14	-11	-9	-8	-8	-9	-11	-14	-18	-22	-26	-31	-34	-38	-40	-42	-43
X= 0.4200	2	1	0	-2	-5	-8	-11	-14	-17	-20	-23	-24	-25	-25	-24	-22	-20	-17	-14	-11	-8	-5	-2	0	1	2
X= 0.4400	-13	-13	-13	-13	-13	-13	-13	-13	-13	-13	-13	-12	-12	-12	-12	-12	-12	-12	-12	-12	-12	-13	-13	-13	-13	-13
X= 0.4600	-9	-9	-9	-9	-8	-8	-7	-6	-6	-5	-5	-4	-4	-4	-4	-5	-5	-6	-6	-7	-8	-8	-9	-9	-9	-9
X= 0.4800	74	72	69	62	54	45	35	25	15	6	-1	-6	-9	-9	-6	-1	6	15	25	35	45	54	62	69	72	74
X= 0.5000	999	979	921	828	707	564	403	251	101	-33	-141	-217	-256	-256	-217	-141	-33	101	251	408	563	706	828	921	979	999
				62	54	45	35	25	15	6	-1	-6	-9	-9	-6	-1	6	15	25	35	45	55	62	69	72	74

(b)

Fig. 5.1 Patterson maps of $W_3V_5O_{20}$

(a) P (uow)

(b) P (uvo)

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$$\rho_o(xyz) = \frac{1}{V}\sum_{hkl} A_{oc} \cos 2\pi(hx+ky+lz) + \frac{1}{V}\sum_{hkl} B_{oc} \sin 2\pi(hx+ky+lz) \quad \ldots\ldots\ldots\ldots\ldots 5.1$$

where $A_{oc}(hkl) = |F_o(hkl)| \cos \alpha_c (hkl)$

$B_{oc}(hkl) = |F_o(hkl)| \sin \alpha_c (hkl)$

$\cos\alpha_c(hkl) = A_c(hkl)/ |F_c(hkl)|$

$\sin\alpha_c(hkl) = B_c(hkl)/ |F_c(hkl)|$

$A_c(hkl) = \sum_n f_n \cos 2\pi(hx_n+ky_n+lz_n)$

$B_c(hkl) = \sum_n f_n \sin 2\pi(hx_n+ky_n+lz_n)$

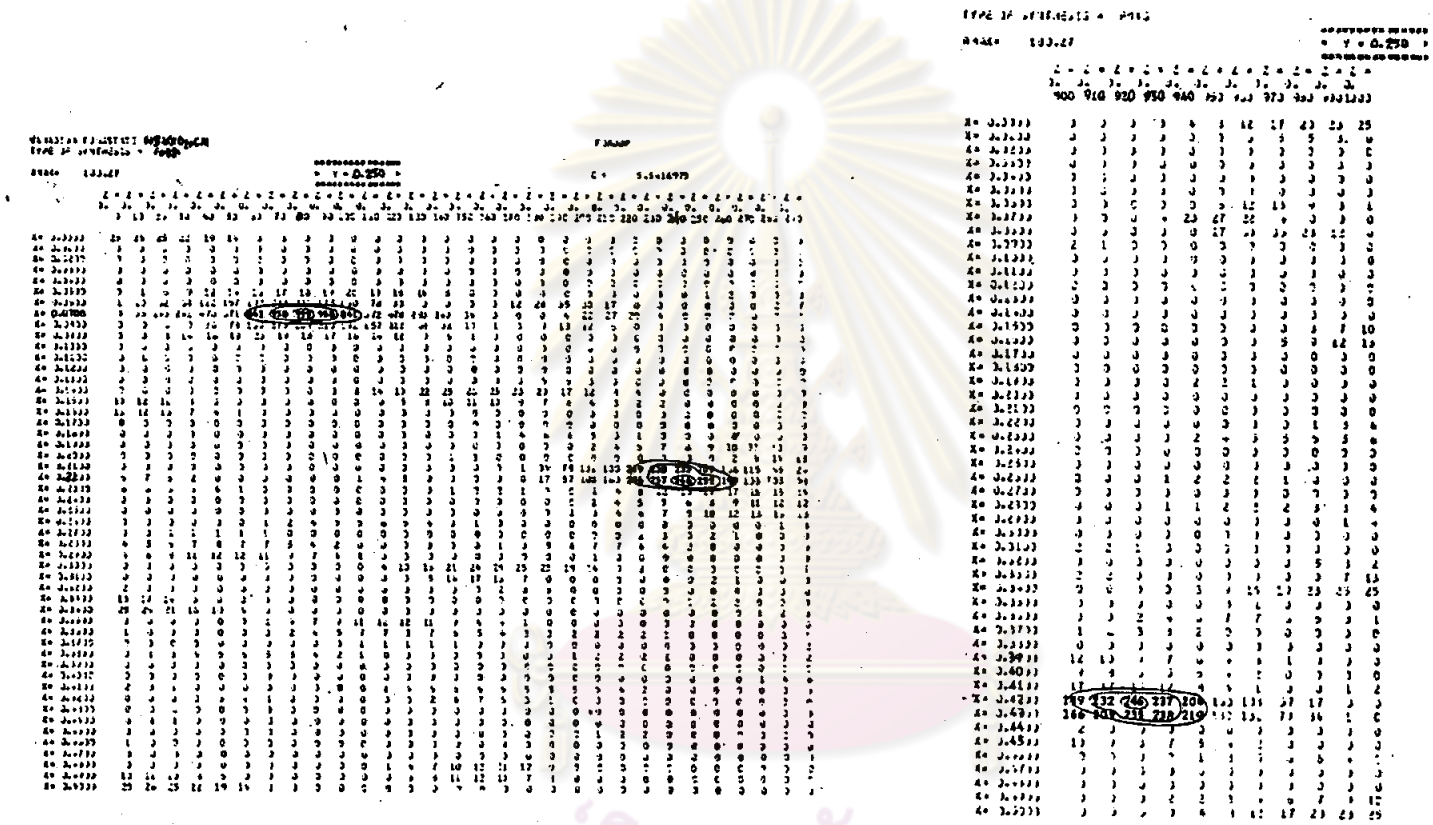
Since the $W_3V_5O_{20}$ crystal has a center of symmetry, Eq. 5.1 gives $B_c = 0$ and

$$\rho_o(xyz) = \frac{1}{V}\sum_{hkl} A_{oc} \cos 2\pi(hx+ky+lz) \quad \ldots\ldots\ldots\ldots\ldots 5.2$$

The sequence of finding the positions of other atoms by F_o synthesis are as follows :

1. The structure factors F_c were calculated by program UPALS using only the positions of tungsten atoms W_1 obtained from the Patterson maps. The temperature factor of 0.33 and the scale factor of 2.0 obtained from the Wilson's plot were also used in the structure factor calculation. The first structure factor calculation yielded the R value of 0.57 for space group C2/m and 0.34

2. The F_o synthesis was then performed by FORDUP program and taking the phase from calculated structure factor based only 8 tungsten atoms W_1 for C2/m and 4 tungsten atoms for Cm. The first electron density map on sections (010) were performed at y=0, 0.25, 0.5 for both



(a)

Fig. 5.2 F_o maps where input "W" = .07 .25 .08, (a),(b),(c) F_o maps with space group Cm, (d),(e),(f) F_o maps with space group C2/m

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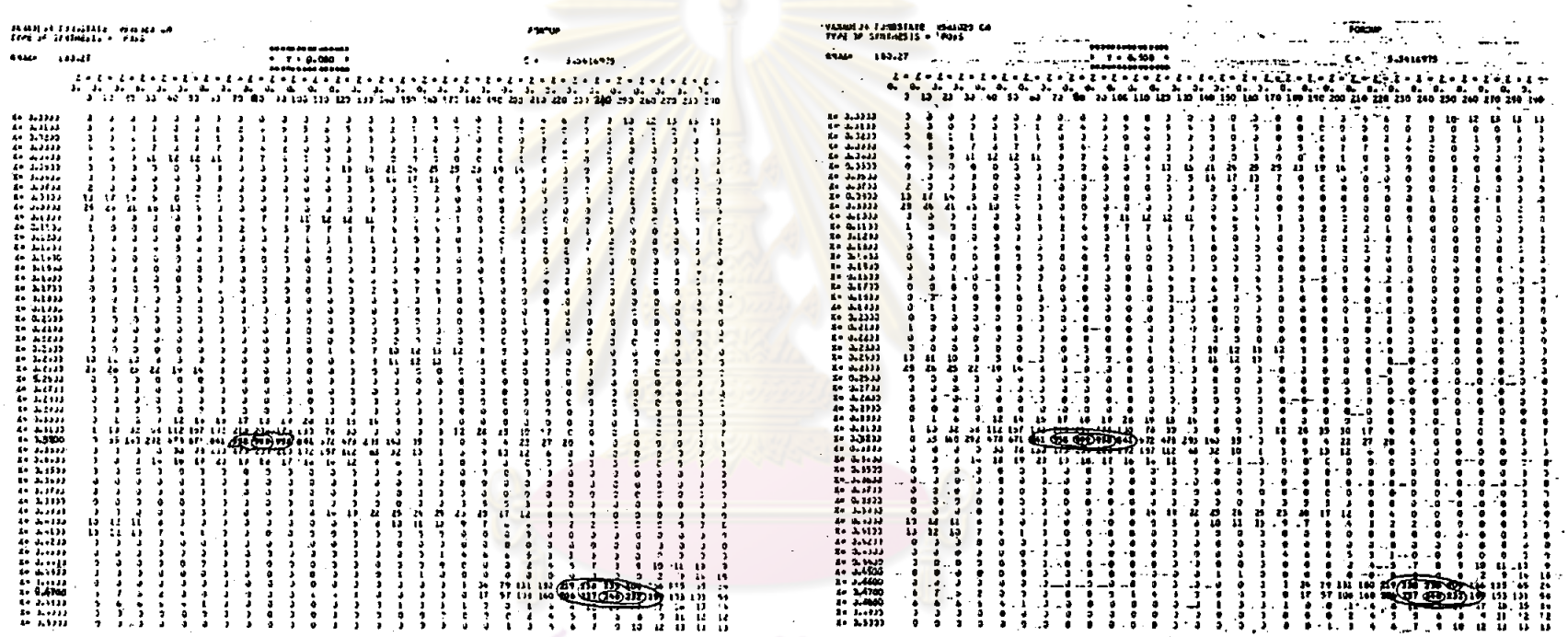

(b)

Fig. 5.2 (continued)

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TYPE OF SYNTHESIS = FUSS

ZMAX= 193.27

Y = 0.900

	Z = 0.900	Z = 0.910	Z = 0.920	Z = 0.930	Z = 0.940	Z = 0.950	Z = 0.960	Z = 0.970	Z = 0.980	Z = 0.990	Z = 1.000
X= 0.0000	0	0	0	0	0	0	0	0	0	0	0
X= 0.0100	0	0	0	1	2	2	2	1	0	0	0
X= 0.0200	0	0	0	0	0	0	0	0	0	0	0
X= 0.0300	0	0	0	1	1	2	2	2	3	3	4
X= 0.0400	0	0	0	0	0	0	0	0	0	1	4
X= 0.0500	0	0	0	0	0	0	0	0	0	0	0
X= 0.0600	0	0	0	0	0	0	0	0	0	0	0
X= 0.0700	2	2	1	0	0	0	0	0	0	0	0
X= 0.0800	0	0	0	0	0	0	0	0	0	0	0
X= 0.0900	1	2	0	0	0	0	0	0	0	0	0
X= 0.1000	0	0	0	0	0	0	0	0	0	0	0
X= 0.1100	0	0	0	0	0	0	0	0	0	0	0
X= 0.1200	1	1	0	0	0	0	0	0	0	0	0
X= 0.1300	0	0	0	0	0	0	0	0	0	0	0
X= 0.1400	12	13	9	7	4	3	1	0	0	0	0
X= 0.1500	4	3	6	5	5	4	2	0	0	0	0
X= 0.1600	17	17	15	12	8	4	1	0	0	1	3
X= 0.1700	193	232	245	237	206	163	101	57	17	3	0
X= 0.1800	166	237	235	238	219	183	121	79	34	1	0
X= 0.1900	2	0	0	0	0	0	0	0	0	0	0
X= 0.2000	13	9	8	7	5	4	2	0	0	0	0
X= 0.2100	0	0	0	0	1	3	5	6	4	1	0
X= 0.2200	0	0	0	0	0	0	0	0	0	0	0
X= 0.2300	0	0	0	0	0	0	0	0	0	0	0
X= 0.2400	0	0	0	0	2	2	3	4	6	7	9	10
X= 0.2500	0	0	0	0	0	4	8	12	17	20	23	25
X= 0.2600	0	0	0	0	0	0	0	1	5	5	3	0
X= 0.2700	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.2800	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.2900	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.3000	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.3100	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.3200	0	0	0	0	0	0	5	12	13	9	5	1
X= 0.3300	0	0	0	0	4	20	27	22	9	3	0	0
X= 0.3400	0	0	0	0	0	6	17	33	35	23	12	0
X= 0.3500	2	1	0	0	0	0	0	0	0	0	0	0
X= 0.3600	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.3700	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.3800	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.3900	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.4000	0	0	0	0	0	0	0	0	0	3	7	10
X= 0.4100	0	0	0	0	0	0	0	0	5	9	12	13
X= 0.4200	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.4300	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.4400	0	0	0	0	0	2	2	1	0	0	0	0
X= 0.4500	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.4600	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.4700	0	0	0	0	0	0	0	0	1	3	5	6
X= 0.4800	0	0	0	0	0	2	4	3	5	5	3	0
X= 0.4900	0	0	0	0	0	0	0	0	0	0	0	0
X= 0.5000	0	0	0	0	0	0	0	0	0	0	0	0

(c)

Fig. 5.2 (condinued)

TYPE OF SYNTHESIS = FUSS

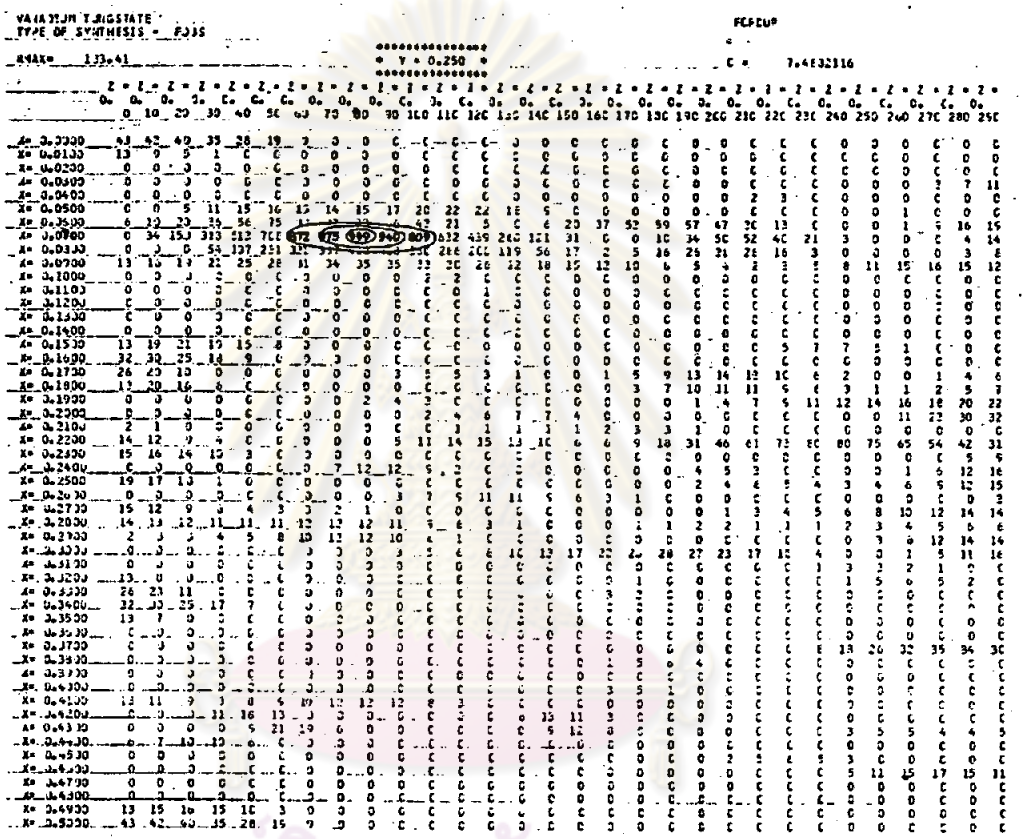
AREA= 133.41

****
* Y = 0.000 *
****

	Z = 0.900	Z = 0.910	Z = 0.920	Z = 0.930	Z = 0.940	Z = 0.950	Z = 0.960	Z = 0.970	Z = 0.980	Z = 0.990	Z = 1.000
X= 0.0100	0	0	0	0	0	0	0	1	10	17	16
X= 0.0200	9	12	12	7	0	0	0	0	0	0	0
X= 0.0300	0	0	0	0	0	0	0	10	14	16	15
X= 0.0400	11	6	0	0	0	0	0	4	9	12	14
X= 0.0500	0	0	0	0	0	0	0	0	0	1	2
X= 0.0600	2	0	0	0	0	0	0	0	0	0	0
X= 0.0700	3	4	2	0	0	0	0	0	0	0	0
X= 0.0800	0	0	0	0	0	0	0	6	16	20	13
X= 0.0900	5	3	0	0	0	0	0	0	10	20	26
X= 0.1000	0	0	0	0	0	0	0	13	25	30	32
X= 0.1100	0	0	0	0	0	3	13	19	21	19	13
X= 0.1200	0	0	0	0	0	0	0	0	0	0	0
X= 0.1300	0	0	0	0	0	0	0	0	0	0	0
X= 0.1400	0	0	0	0	0	0	0	0	0	0	0
X= 0.1500	0	0	0	0	0	0	0	0	0	0	0
X= 0.1600	2	0	1	0	0	0	0	0	0	0	0
X= 0.1700	33	35	35	34	31	28	25	21	19	16	13
X= 0.1800	309	340	339	325	312	321	232	137	54	0	0
X= 0.1900	809	940	999	975	872	708	513	319	153	34	0
X= 0.2000	45	56	61	61	63	75	56	35	20	10	6
X= 0.2100	20	17	15	14	15	16	15	11	5	0	0
X= 0.2200	0	0	0	0	0	0	0	0	0	0	0
X= 0.2300	0	0	0	0	0	0	0	0	0	0	0
X= 0.2400	0	0	0	0	0	0	0	0	0	0	0
X= 0.2500	0	0	0	0	0	0	0	1	5	9	12
X= 0.2600	0	0	0	0	5	19	28	35	40	42	43
X= 0.2700	0	0	0	0	0	3	10	15	16	15	13
X= 0.2800	0	0	0	0	0	0	0	0	0	0	0
X= 0.2900	0	0	0	0	0	0	0	0	0	0	0
X= 0.3000	0	0	0	0	0	0	0	0	0	0	0
X= 0.3100	0	0	0	0	0	0	0	11	10	7	6
X= 0.3200	0	0	0	0	16	21	0	0	0	0	0
X= 0.3300	0	0	0	0	0	15	13	11	0	0	0
X= 0.3400	8	12	13	12	10	9	0	8	9	11	12
X= 0.3500	0	0	0	0	0	0	0	0	0	0	0
X= 0.3600	0	0	0	0	0	0	0	0	0	0	0
X= 0.3700	0	0	0	0	0	0	0	0	0	0	0
X= 0.3800	0	0	0	0	0	0	0	0	0	0	0
X= 0.3900	0	0	0	0	0	0	0	0	0	0	0
X= 0.4000	0	0	0	0	0	0	0	0	0	7	13
X= 0.4100	0	0	0	0	0	0	7	17	25	30	32
X= 0.4200	0	0	0	0	0	0	0	0	11	23	26
X= 0.4300	0	0	0	0	0	0	0	0	0	0	13
X= 0.4400	0	0	0	0	0	0	0	0	0	0	0
X= 0.4500	5	3	0	0	0	0	0	0	0	0	0
X= 0.4600	6	10	12	12	10	8	5	4	3	3	2
X= 0.4700	9	11	12	12	12	11	11	11	12	13	14
X= 0.4800	0	0	1	2	3	3	4	5	9	12	15
X= 0.4900	7	3	0	0	0	0	0	0	0	0	0
X= 0.5000	0	0	0	0	0	0	0	1	10	17	19

(d)

Fig. 5.2 (continued)



(e)

Fig. 5.2 (continued)

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TYPE OF SYNTHESIS = FUBS

MAX= 135.41

* Y = 0.800 *

	Z = 0. 900	Z = 0. 910	Z = 0. 920	Z = 0. 930	Z = 0. 940	Z = 0. 950	Z = 0. 960	Z = 0. 970	Z = 0. 980	Z = 0. 990	Z = 0. 1000
X= 0.0000	0	0	0	0	0	0	0	1	10	17	19
X= 0.0100	9	12	12	7	0	0	0	0	0	0	0
X= 0.0200	0	0	0	0	0	0	3	10	14	16	15
X= 0.0300	11	5	0	0	0	0	0	4	9	12	14
X= 0.0400	0	0	0	0	0	0	0	0	0	1	2
X= 0.0500	2	0	0	0	0	0	0	0	0	0	0
X= 0.0600	3	4	2	0	0	0	0	0	0	0	0
X= 0.0700	0	0	0	0	0	0	0	6	15	20	19
X= 0.0800	5	3	0	0	0	0	0	0	10	20	26
X= 0.0900	0	0	0	0	0	0	0	18	25	30	32
X= 0.1000	0	0	0	0	0	8	15	19	21	19	19
X= 0.1100	0	0	0	0	0	0	0	0	0	0	0
X= 0.1200	0	0	0	0	0	0	0	0	0	0	0
X= 0.1300	0	0	0	0	0	0	0	0	0	0	0
X= 0.1400	0	0	0	0	0	0	0	0	0	0	0
X= 0.1500	2	0	0	0	0	0	0	0	0	0	0
X= 0.1600	33	35	35	34	31	28	25	21	17	16	13
X= 0.1700	360	431	461	437	421	232	137	54	0	0	0
X= 0.1800	809	940	999	975	872	708	513	319	153	34	0
X= 0.1900	43	69	77	72	68	75	55	36	20	10	6
X= 0.2000	20	17	15	14	15	16	15	11	5	0	0
X= 0.2100	0	0	0	0	0	0	0	0	0	0	0
X= 0.2200	0	0	0	0	0	0	0	0	0	0	0
X= 0.2300	0	0	0	0	0	0	0	0	0	0	0
X= 0.2400	0	0	0	0	0	0	0	1	5	9	13
X= 0.2500	0	0	0	0	9	19	28	35	40	42	43
X= 0.2600	0	0	0	0	0	8	14	15	16	15	13
X= 0.2700	0	0	0	0	0	0	0	0	0	0	0
X= 0.2800	0	0	0	0	0	0	0	0	0	0	0
X= 0.2900	0	0	0	0	0	0	0	0	0	0	0
X= 0.3000	0	0	0	0	0	0	0	0	0	0	0
X= 0.3100	0	0	0	0	0	0	6	10	10	7	6
X= 0.3200	0	0	0	6	19	21	7	0	0	0	0
X= 0.3300	0	0	0	0	0	13	13	11	0	0	0
X= 0.3400	8	12	13	12	10	6	3	3	9	11	13
X= 0.3500	0	0	0	0	0	0	0	0	0	0	0
X= 0.3600	0	0	0	0	0	0	0	0	0	0	0
X= 0.3700	0	0	0	0	0	0	0	0	0	0	0
X= 0.3800	0	0	0	0	0	0	0	0	0	0	0
X= 0.3900	0	0	0	0	0	0	0	0	0	0	0
X= 0.4000	0	0	0	0	0	0	0	0	0	7	13
X= 0.4100	0	0	0	0	0	0	7	17	25	30	32
X= 0.4200	0	0	0	0	0	0	0	0	11	23	26
X= 0.4300	0	0	0	0	0	0	0	0	0	0	13
X= 0.4400	0	0	0	0	0	0	0	0	0	0	0
X= 0.4500	5	3	0	0	0	0	0	0	0	0	0
X= 0.4600	6	10	12	12	10	8	5	4	3	3	2
X= 0.4700	9	11	12	12	12	11	11	11	12	13	14
X= 0.4800	0	0	1	2	3	5	4	6	9	12	15
X= 0.4900	7	3	0	0	0	0	0	0	0	0	0
X= 0.5000	0	0	0	0	0	0	0	1	10	17	19

(f)

Fig. 5.2 (continued)

space groups. The F_o synthesis of these two space groups were prepared only in one asymmetric unit which was one-fourth of cell volume, i.e. x = 0-0.5, z = 0-1.0. These maps were shown in Fig. 5.2

The maximum electron density is proportional to the atomic number of the atom. For $W_3V_5O_{20}$ the atomic number 74 corresponds to electron density peak 999, therefore the atomic number 23 and 8 should approximately correspond to peaks of height 311 and 108 respectively.

By considering the electron density peak from the map it was found 28 metal atoms (Table 5.6) for space group "Cm" which are much more than the number of metals of $W_3V_5O_{20}$ (16 atoms) in a unit cell. So this space group is not fit for these intensity data. For space group C2/m only 3 peaks (Table 5.6) are obtained. Thus space group C2/m was used for later calculations.

Table 5.6 Coordinates of peaks obtained from ρ_o maps of space group of C2/m and C2 were shown.

space group	peak height	coordinates	calculated atoms from F_o map
Cm	999	0.07,0.25,0.08	4
	999	0.32,0.50,0.08	2
	999	0.07,0.75,0.08	4
	246	0.47,0.00,0.24	2
	246	0.22,0.25,0.24	4
	246	0.42,0.25,0.92	4
	246	0.47,0.50,0.24	2
	246	0.17,0.50,0.92	2
	246	0.22,0.75,0.24	4
C2/m	999	0.07,0.25,0.08	8
	999	0.18,0.00,0.92	4
	999	0.18,0.50,0.92	4

From the F_o map better coordinates of atoms were obtained. The shifted atomic positions were determined by the Booth's method.

Table 5.7 $\Delta\rho$ as a function of x coordinate in the Booth's method

x	0	1	2
$\Delta\rho$	0	ρ_1	ρ_2

$\rho_1 \geq \rho_2 \geq 0$

From Table 4.6 the highest peak obtained from the electron density map is at the point x=1. $\Delta\rho'$ is the difference between electron density of each point and that of the lowest point, i.e. at x=0. The electron density is assumed to be given by the equation

$$\rho = ax^2+bx \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad 5.3$$

Substituting the value of x corresponding to ρ_1 and ρ_2 from table 5.7 Eq. 5.3 gives

$$a = \frac{\rho_2-2\rho_1}{2}$$

$$b = -\left(\frac{\rho_2-4\rho_1}{2}\right)$$

The position of the maximum electron density is obtained by differentiating Eq. 5.3 and equating the result to zero

$$x_m = \frac{-b}{2a}$$

$$= \frac{\rho_2-4\rho_1}{2\rho_2-4\rho_1}$$

$$= \frac{(\rho_2/\rho_1) - 4}{(2\rho_2/\rho_1) - 4} \quad \ldots\ldots\ldots\ldots\ldots \quad 5.4$$

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where x_m is the distance to be shifted from x=0. The y and z coordinates are also calculated by the same procedure as the x coordinate.

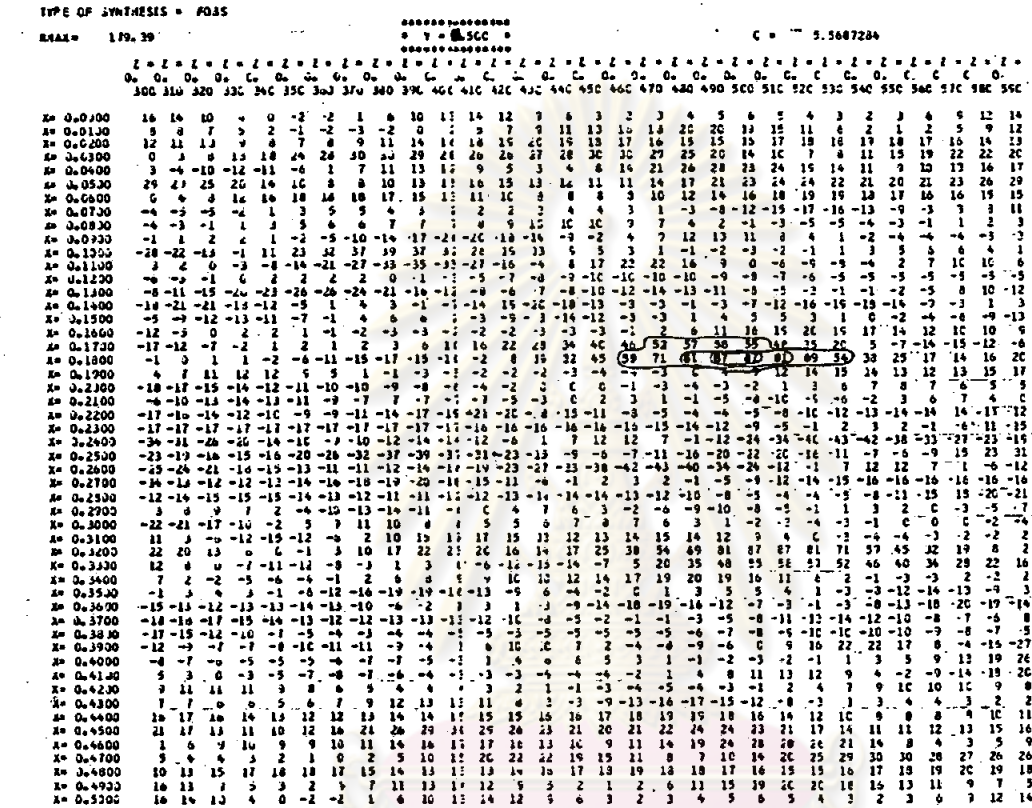
The coordinates of atoms obtained from the first electron density map are listed in Table 5.8

Table 5.8 The coordinate of atoms obtained from the first electron density map and refined by Booth's method.

peak height	atomic coordinate	position in C2/m
999	0.071,0.25,0.078	8j
999	0.179,0.50,0.922	4i
999	0.179,0.00,0.922	4i

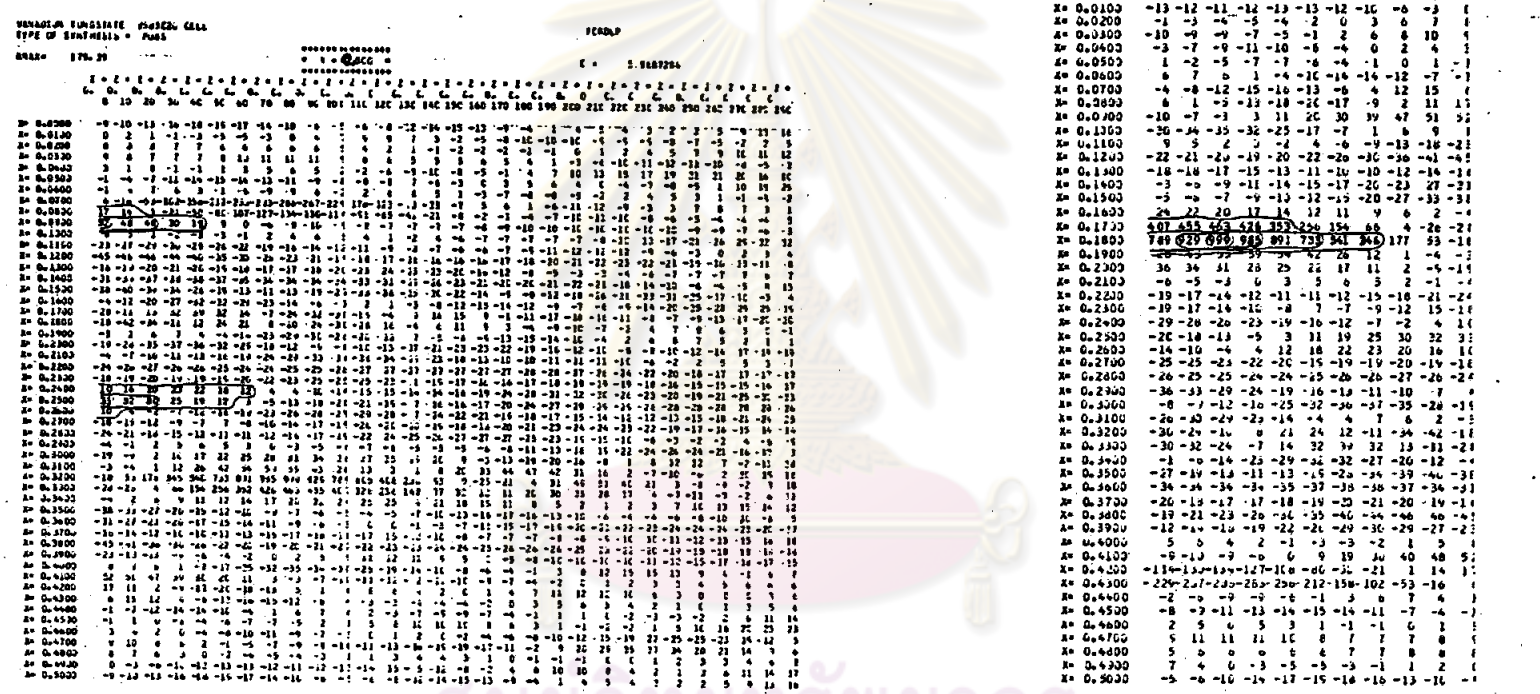
3. All of the three 999 peaks were first assigned as positions of "W". The calculations then were performed as in step2. The second F_o map obtained show that no peaks corresponded to "V" positions. By considering the number of atoms in a unit cell of 6 W.,10 V and 20 oxygen atoms, the assignments of the three peaks were 8 W_1 in 8j position (.071, .25, .078); 4 V_2 in 4i (.179, 0, .922) and 4 W_2 in 4i (.179, .5, .922). The structure factor calculation obtained from these assignments gave the R value of 0.311. However the number of "W" positions in this assignment was more than that expected in $W_3V_5O_{20}$ and that of "V" positions was lower than that expected in $W_3V_5O_{20}$. Thus the position (.071, .25, .078) must be occupied by both W and V and the occupancy factors of "W" and of "V" should be 0.25 and 0.75 respectively.

4. The same procedure was repeated as "step2" to obtain the third F_o maps (Fig. 5.4) using (W_1V_1) in 8j (0.71, 0.25, 0.078) with



(a)

Fig. 5.3 (a),(b),(c),(d),(e),(f) F_o maps where input "W_1", "V_1", "W_2" "V_2", with space group C2/m



(b)

Fig. 5.3 (continued)

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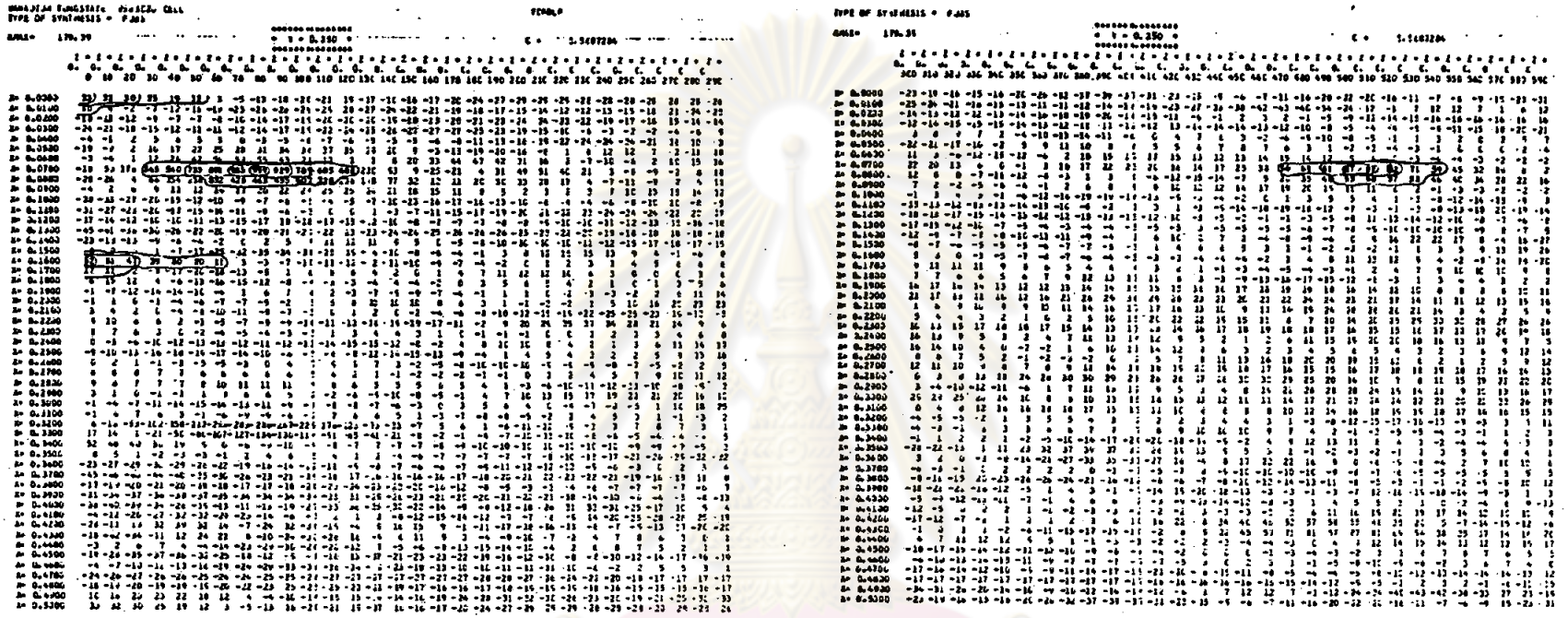

(c)

Fig. 5.3 (continued)

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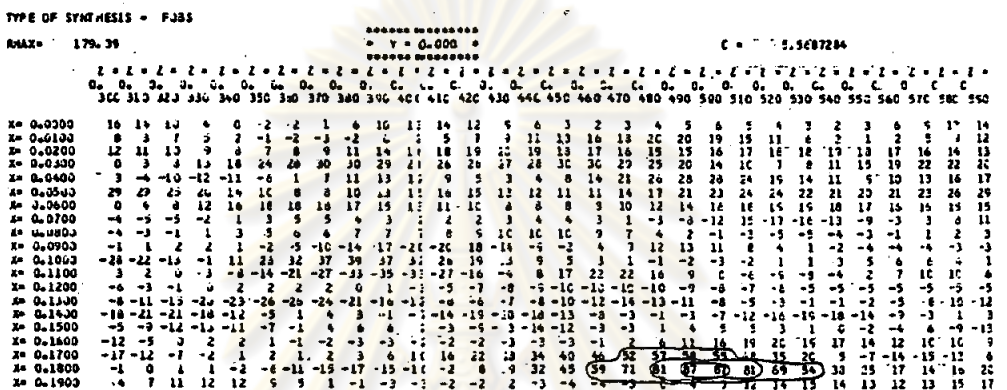

(d)

Fig. 5.3 (continued)

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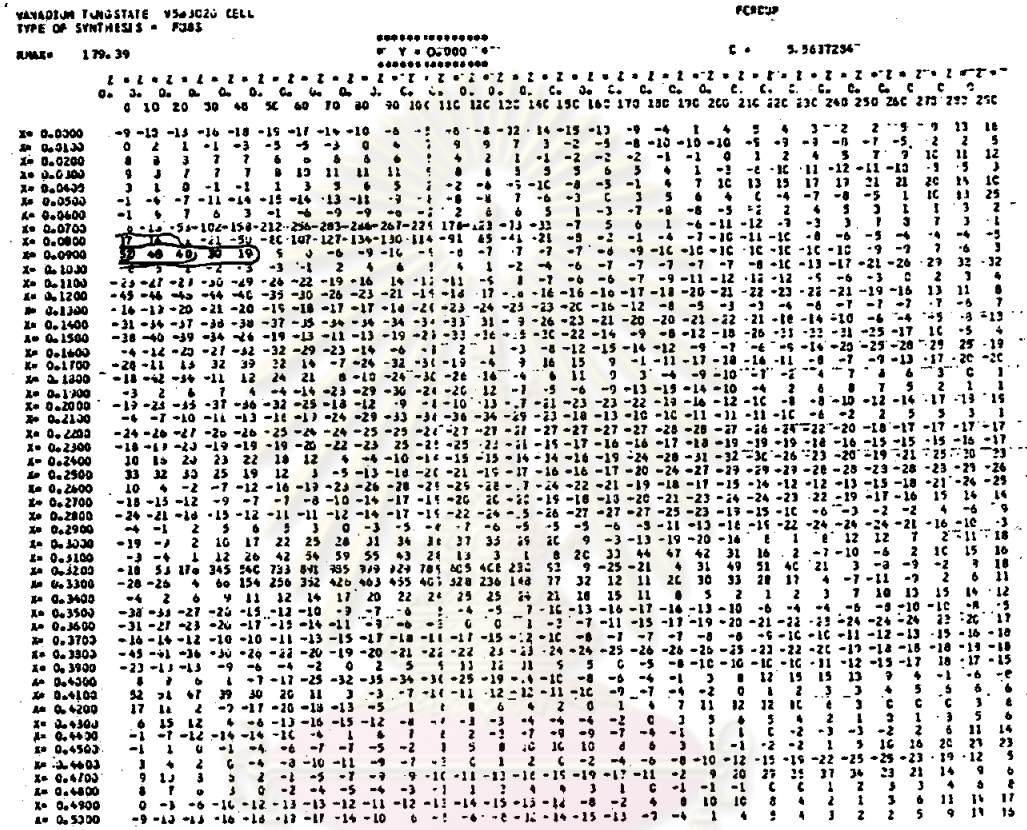

(e)

Fig. 5.3 (continued)

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TYPE OF SYNTHESIS = FOBS

RMAX= 179.39

Y = 0.500

	Z = 0.900	Z = 0.910	Z = 0.920	Z = 0.930	Z = 0.940	Z = 0.950	Z = 0.960	Z = 0.970	Z = 0.980	Z = 0.990	Z = 1.000
X= 0.0000	-5	-8	-10	-14	-17	-19	-18	-16	-13	-10	-9
X= 0.0100	-13	-12	-11	-12	-13	-13	-12	-10	-8	-5	0
X= 0.0200	-1	-3	-4	-5	-4	-2	0	3	6	7	8
X= 0.0300	-10	-9	-9	-7	-5	-1	2	6	8	10	9
X= 0.0400	-3	-7	-9	-11	-10	-8	-4	0	2	4	3
X= 0.0500	1	-2	-5	-7	-7	-6	-4	-1	0	1	1
X= 0.0600	6	7	6	1	-4	-10	-14	-14	-12	-7	-1
X= 0.0700	-4	-3	-12	-15	-16	-13	-6	4	12	15	14
X= 0.0800	6	1	-5	-13	-18	-20	-17	-9	2	11	17
X= 0.0900	-10	-7	-3	3	11	20	30	39	47	51	52
X= 0.1000	-30	-34	-35	-32	-25	-17	-7	1	6	9	8
X= 0.1100	9	5	2	0	-2	-4	-6	-9	-13	-18	-23
X= 0.1200	-22	-21	-20	-19	-20	-22	-26	-33	-36	-41	-45
X= 0.1300	-18	-18	-17	-15	-13	-11	-10	-10	-12	-14	-16
X= 0.1400	-3	-6	-9	-11	-14	-15	-17	-20	-23	-27	-31
X= 0.1500	-5	-6	-7	-9	-10	-12	-15	-20	-27	-33	-38
X= 0.1600	24	22	20	17	14	12	11	9	6	2	-4
X= 0.1700	407	455	463	426	353	256	154	66	4	-26	-28
X= 0.1800	780	929	999	985	891	733	541	340	177	53	-18
X= 0.1900	28	43	55	59	54	42	26	12	1	-4	-3
X= 0.2000	36	34	31	28	25	22	17	11	2	9	19
X= 0.2100	-6	-5	-3	0	3	5	5	5	2	-1	-4
X= 0.2200	-19	-17	-14	-12	-11	-11	-12	-15	-18	-21	-24
X= 0.2300	-19	-17	-14	-10	-8	-7	-7	-9	-12	-15	-18
X= 0.2400	-29	-28	-26	-23	-19	-16	-12	-7	-2	4	10
X= 0.2500	-20	-18	-13	-5	3	11	19	25	30	32	33
X= 0.2600	-14	-10	-4	4	12	18	22	23	20	16	10
X= 0.2700	-25	-25	-23	-22	-20	-19	-19	-19	-20	-19	-18
X= 0.2800	-26	-25	-25	-24	-24	-25	-26	-26	-27	-26	-24
X= 0.2900	-36	-33	-29	-24	-19	-16	-13	-11	-10	-7	-4
X= 0.3000	-8	-9	-12	-18	-25	-32	-36	-37	-35	-28	-19
X= 0.3100	-26	-30	-29	-23	-14	-4	4	7	6	2	-3
X= 0.3200	-30	-24	-10	8	21	24	12	-11	-34	-42	18
X= 0.3300	-30	-32	-24	-7	14	32	39	32	13	-11	-28
X= 0.3400	-1	-6	-14	-23	-29	-32	-32	-27	-20	-12	-4
X= 0.3500	-27	-19	-13	-11	-13	-19	-26	-34	-39	-40	-38
X= 0.3600	-34	-34	-34	-34	-35	-37	-38	-38	-37	-34	-31
X= 0.3700	-20	-18	-17	-17	-18	-19	-20	-21	-20	-19	-16
X= 0.3800	-19	-21	-23	-26	-30	-35	-40	-44	-46	-46	-45
X= 0.3900	-12	-14	-16	-19	-22	-26	-29	-30	-29	-27	-23
X= 0.4000	5	3	4	2	-1	-3	-3	-2	1	5	8
X= 0.4100	-9	-10	-9	-6	0	9	19	30	40	48	52
X= 0.4200	-114	-130	-134	-127	-108	-80	-50	-21	1	14	17
X= 0.4300	-229	-267	-266	-263	-256	-212	-158	-102	-53	-16	6
X= 0.4400	-2	-6	-9	-9	-6	-1	3	6	7	4	-1
X= 0.4500	-8	-9	-11	-13	-14	-15	-14	-11	-7	4	-1
X= 0.4600	2	5	6	5	3	1	-1	-1	0	1	3
X= 0.4700	9	11	11	11	10	8	7	7	7	8	9
X= 0.4800	5	6	6	6	6	6	7	7	8	8	8
X= 0.4900	7	5	0	-3	-5	-5	-3	-1	1	2	0
X= 0.5000	-5	-8	-10	-14	-17	-19	-18	-16	-13	-10	-9

(f)

Fig. 5.3 (continued)

occupancy factor of 0.25 and 0.75 for W_1 and V_1 respectively, W_2 in 4i (.179, .5, .922), V_2 in 4i (0.179, 0.5, 0.922). These gave the positions (8 coordinates) of 8 independent oxygen atoms. The better atomic coordinates were made by Booth's method as tabulated in Table 5.9

Table 5.9 Coordinates of all atoms of $W_3V_5O_{20}$ in an asymmetric unit.

Position in C 2/m	atom	peak height	atomic coordinate
8j	W_1 (V_1)	999	0.071 ,0.25 ,0.078
4i	W_2	999	0.179 ,0.50 ,0.922
4i	V_2	999	0.179 ,0.00 ,0.922
4g	O_1	33	0.00 ,0.25 ,0.00
4i	O_2	52	0.0895,0.00 ,0.00
8j	O_3	52	0.161 ,0.25 ,0.00
4i	O_4	54	0.0895,0.50 ,0.00
4i	O_5	33	0.250 ,0.50 ,0.00
8j	O_6	87	0.073 ,0.25 ,0.507
4i	O_7	87	0.177 ,0.50 ,0.493
4i	O_8	87	0.177 ,0.00 ,0.493

Least Squares Refinement

The structure was finally refined by the full matrix least squares method using the UPALS program with Cruickshank[35] weight scheme. The individual isotropic temperature factors were assigned as 0.33 obtained from Wilson's plot for every atoms. The refinement of structure in this work was the refinement of atomic positions and isotropic temperature factors. The 469 observed unique reflections

were used to refine atomic positions and isotropic temperature factors. The scale factors, k_1,k_2 for hol and h21 respectively were refined in every refinement.

The refinements of atomic positions were separated into 4 steps as follows.

At first only coordinates of W_1 and V_1 were simultaneously refined by resetting V_1 and the other atomic positions were fixed. Three cycles of refinement led to the value of R=0.136 Second step, the coordinates of W_2, W_1 and V_1 were simultaneously refined by fixing the other atomic positions. Three cycles of refinement were made. This led to the better k value of 0.114. Third step only the coordinates of V_2 was refined by fixing other atomic positions. Three cycles of refinement led to the value of R=0.113. The forth step, coordinates of 8 oxygen atoms were refined. Firstly only the coordinates of an oxygen atom O_3 in 8j position was refined and three cycles of refinement were made. Then the atomic coordinates of O_3 and O_6 both atoms in 8j position were simultaneously refined and again with three cycles. The coordinates of independent oxygen atoms simultaneously refined were added one at each refinement until all coordinates of eight independent atoms were simultaneously refined except O_1. Because the position of O_1 is 0., 0.255, 0. and y coordinate can not be refined due to limited data. Again three cycles of refinement were made. The final R value was 0.113.

The isotropic temperature factor of each individual atoms was refined in later stage by fixing all of atomic positions and the isotropic temperature factors of other atoms. Three cycles for each refinement was made. The refinement of isotropic temperature factors was also

separated in 4 steps.

Isotropic temperature factor of W_1 was refined first by resetting isotropic temperature factor of V_1. The second step was refinement of W_2. The third was of V_2 and finally refinement of overall isotropic temperature factors of all eight oxygen atoms were made simultaneously. The R values of 4 step were 0.112, 0.114, 0.113 and 0.112 respectively.

The final parameters are listed in Table 5.10. The observed and calculated structure factors are listed in Table 5.11.

Table 5.10 The final atomic coordinates, thermal parameters and their standard deviations in the last digits given within parentheses.

Atom	X	Y	Z	B Å^2
W_1 (V_1)	0.0744 (2)	0.25	0.0867 (17)	0.472 (44)
W_2	0.1793 (1)	0.50	0.9285 (8)	0.278 (4)
V_2	0.1755 (4)	0.	0.9229 (24)	0.260 (3)
O_1	0.0	0.250	0.0	0.320 (4)
O_2	0.087271	0.0	0.01451	0.320 (4)
O_3	0.1590 (12)	0.25	0.0189 (48)	0.320 (4)
O_4	0.0929 (17)	0.50	0.0262 (150)	0.320 (4)
O_5	0.2508 (13)	0.50	0.9616 (76)	0.320 (4)
O_6	0.0776 (9)	0.25	0.5070 (58)	0.320 (4)
O_7	0.1767 (17)	0.50	0.4712 (111)	0.320 (4)
O_8	0.181312	0.0	0.5175 (106)	0.320 (4)

Table 5.11 The observed and calculated structure factors of 469 reflections

H	K	L	YOE	YC
8	0	0	443.91	535.48
12	0	0	451.09	503.49
16	0	0	198.66	218.19
20	0	0	365.48	356.22
24	0	0	49.86	27.44
28	0	0	236.40	278.77
32	0	0	99.01	90.92
36	0	0	152.70	153.04
40	0	0	153.59	159.64
44	0	0	67.84	72.16
48	0	0	119.67	139.99
56	0	0	89.47	104.21
60	0	0	40.72	59.83
64	0	0	34.83	31.56
0	0	2	403.88	450.34
0	0	3	46.77	57.18
0	0	4	64.73	51.06
0	0	5	245.52	194.45
0	0	6	195.93	170.84
0	0	7	189.42	163.84
0	0	8	95.84	78.06
0	0	9	43.21	32.21
0	0	10	31.82	29.55
4	0	1	375.26	372.30
4	0	2	452.65	488.18
4	0	3	377.80	372.15
4	0	4	260.87	268.02
4	0	5	109.67	99.88
4	0	7	132.70	94.59
4	0	8	134.47	111.09
4	0	9	134.69	114.91
4	0	10	81.98	74.86
8	0	1	176.05	182.51
8	0	2	43.11	57.71
8	0	3	187.29	191.52
8	0	4	230.15	224.61
8	0	5	274.85	260.82
8	0	6	182.99	184.40
8	0	7	133.06	121.91
8	0	10	66.79	59.60
12	0	1	476.47	482.61
12	0	2	449.15	507.47
12	0	3	236.99	234.79
12	0	4	153.28	147.90
12	0	7	153.38	134.46
12	0	8	106.78	108.51
12	0	9	87.79	90.68
12	0	10	35.13	37.15
16	0	1	40.15	17.26
16	0	2	165.33	160.36

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Table 5.11 (continued)

H	K	L	YOE	YC
16	0	3	239.68	267.27
16	0	4	280.02	264.11
16	0	5	233.27	214.65
16	0	6	151.15	127.84
16	0	9	67.84	64.17
16	0	10	67.32	68.95
20	0	1	273.65	275.63
20	0	2	250.46	247.13
20	0	3	61.65	60.38
20	0	5	152.32	139.30
20	0	6	147.38	145.46
20	0	7	163.98	151.82
20	0	8	108.92	95.41
20	0	9	68.36	55.27
24	0	1	170.03	157.39
24	0	2	240.53	262.42
24	0	3	264.77	260.36
24	0	4	237.67	227.10
24	0	5	126.06	127.49
24	0	6	58.10	52.83
24	0	8	63.40	58.38
24	0	9	76.97	79.03
24	0	10	42.15	58.22
28	0	1	203.12	173.67
28	0	2	103.10	94.82
28	0	3	52.60	47.18
28	0	4	101.45	108.73
28	0	5	128.70	159.47
28	0	6	126.60	138.93
28	0	7	103.46	105.60
28	0	8	59.71	51.90
32	0	1	154.70	145.42
32	0	2	225.43	198.80
32	0	3	131.55	143.77
32	0	4	98.68	100.22
32	0	7	77.18	88.75
32	0	8	78.22	81.19
32	0	9	76.57	75.19
36	0	1	61.17	56.11
36	0	3	95.96	109.69
36	0	4	122.53	142.47
36	0	5	126.85	133.23
36	0	6	97.19	100.80
36	0	7	68.62	46.82
40	0	1	141.14	158.81
40	0	2	144.35	157.36
40	0	3	79.67	80.80
40	0	6	54.49	67.54
40	0	7	73.70	83.36
40	0	8	59.13	65.08
44	0	2	51.69	67.35
44	0	3	71.96	91.12
44	0	4	79.62	100.32
44	0	5	66.22	62.51
44	0	6	41.23	34.75

ศูนย์วิทยทรัพยากร
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Table 5.11 (continued)

H	K	L	YOE	YC
48	0	1	106.16	111.23
48	0	2	76.47	76.33
48	0	5	40.48	64.34
48	0	6	51.36	71.83
48	0	7	44.68	58.66
52	0	1	45.82	68.21
52	0	2	68.08	104.01
52	0	3	65.88	90.37
52	0	4	62.16	76.21
52	0	5	40.37	30.27
56	0	1	60.89	70.22
56	0	5	32.06	46.48
56	0	6	25.47	46.43
60	0	1	48.39	72.52
60	0	2	47.16	77.60
60	0	3	39.52	53.41
60	0	4	33.09	29.72
64	0	3	18.55	45.17
-4	0	1	232.64	145.64
-4	0	2	419.61	296.02
-4	0	3	529.54	370.75
-4	0	4	427.96	306.11
-4	0	5	274.57	208.13
-4	0	6	94.85	87.06
-4	0	8	94.37	76.82
-4	0	9	108.32	102.62
-4	0	10	102.45	95.07
-8	0	1	389.69	427.45
-8	0	2	385.18	441.76
-8	0	3	153.66	165.97
-8	0	4	49.77	54.59
-8	0	5	138.95	120.79
-8	0	6	185.71	148.91
-8	0	7	218.91	186.36
-8	0	8	156.23	127.38
-8	0	9	107.34	91.58
-12	0	1	185.61	170.98
-12	0	2	25.09	19.71
-12	0	3	207.18	211.17
-12	0	4	217.57	225.96
-12	0	5	275.90	236.73
-12	0	6	166.27	140.15
-12	0	7	78.83	71.95
-12	0	9	78.36	58.66
-12	0	10	88.33	87.28
-16	0	1	286.34	315.32
-16	0	2	316.68	377.24
-16	0	3	235.22	264.22
-16	0	4	164.03	164.23
-16	0	6	76.04	69.54
-16	0	7	162.71	140.14
-16	0	8	159.47	132.91
-16	0	9	141.48	118.07
-16	0	10	71.44	65.46
-20	0	1	185.10	183.38

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Table 5.11 (continued)

H	K	L	YOE	YC
-20	0	2	95.74	99.84
-20	0	3	103.72	110.40
-20	0	4	122.88	161.95
-20	0	5	224.43	238.82
-20	0	6	205.18	183.08
-20	0	7	177.51	151.05
-20	0	8	73.06	55.39
-20	0	10	42.19	48.89
-24	0	1	116.27	134.92
-24	0	2	213.99	224.31
-24	0	3	288.50	249.70
-24	0	4	206.39	197.62
-24	0	5	132.43	110.81
-24	0	7	69.02	58.86
-24	0	8	109.63	99.35
-24	0	9	118.78	106.13
-24	0	10	92.13	86.57
-28	0	1	212.79	231.82
-28	0	2	211.09	196.74
-28	0	3	59.47	43.78
-28	0	5	153.80	143.35
-28	0	6	185.83	146.99
-28	0	7	179.44	158.22
-28	0	8	123.22	95.68
-28	0	9	68.03	58.40
-32	0	1	39.37	49.66
-32	0	2	133.92	129.87
-32	0	3	263.36	217.56
-32	0	4	277.56	206.54
-32	0	5	201.68	183.62
-32	0	6	120.72	98.98
-32	0	7	38.38	36.59
-32	0	8	79.13	34.94
-32	0	9	70.88	62.11
-36	0	1	180.54	187.85
-36	0	2	204.86	196.45
-36	0	3	162.62	125.13
-36	0	4	91.06	59.21
-36	0	6	79.83	81.43
-36	0	7	139.32	118.72
-36	0	8	125.62	100.44
-36	0	9	92.52	79.68
-40	0	1	60.10	70.05
-40	0	3	108.29	95.26
-40	0	4	151.25	123.37
-40	0	5	184.01	154.45
-40	0	6	145.02	111.03
-40	0	7	85.63	79.59
-44	0	1	139.21	140.67
-44	0	2	160.09	171.05
-44	0	3	170.08	163.78
-44	0	4	136.00	120.42
-44	0	5	71.88	56.37
-44	0	7	55.04	53.51
-44	0	8	74.26	74.19

ศูนย์วิทยทรัพยากร
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Table 5.11 (continued)

H	K	L	YOE	YC
-48	0	1	94.78	109.10
-48	0	2	68.00	74.50
-48	0	4	50.04	46.95
-48	0	5	106.10	97.86
-48	0	6	102.21	93.45
-48	0	7	97.43	88.34
-52	0	1	37.73	35.84
-52	0	2	62.61	75.29
-52	0	3	93.49	108.69
-52	0	4	86.20	98.50
-52	0	5	72.52	76.12
-52	0	6	29.31	28.82
-56	0	1	85.20	113.03
-56	0	2	83.27	101.53
-56	0	3	52.34	59.22
-56	0	5	35.06	33.43
-56	0	6	52.21	56.51
-60	0	3	53.19	59.36
-60	0	4	57.31	71.21
-60	0	5	58.90	74.48
-64	0	1	40.00	58.91
-64	0	2	45.32	65.33
-64	0	3	35.41	56.43
10	2	0	102.80	98.59
14	2	0	510.38	527.17
18	2	0	121.06	104.65
22	2	0	268.65	310.37
26	2	0	272.26	277.61
30	2	0	131.42	129.32
34	2	0	223.70	238.71
38	2	0	53.39	47.40
42	2	0	159.24	167.54
46	2	0	77.12	85.71
50	2	0	68.90	62.38
54	2	0	82.86	96.77
58	2	0	37.67	22.11
62	2	0	47.14	65.60
2	2	2	143.83	110.68
2	2	3	305.97	295.19
2	2	4	300.95	283.66
2	2	5	294.34	254.81
2	2	6	177.71	149.62
2	2	7	71.76	63.96
2	2	9	69.75	63.50
2	2	10	85.52	81.51
6	2	1	351.89	314.90
6	2	2	307.38	339.05
6	2	3	94.19	79.80
6	2	5	180.55	162.87
6	2	6	190.76	171.60
6	2	7	206.46	191.83
6	2	8	136.53	124.39
6	2	9	106.10	82.60
10	2	1	162.34	138.14
10	2	2	295.11	289.71

ศูนย์วิทยทรัพยากร
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Table 5.11 (continued)

H	K	L	YOE	YC
10	2	3	318.30	317.11
10	2	4	268.39	269.21
10	2	5	172.96	161.75
10	2	6	68.13	61.84
10	2	7	44.33	33.98
10	2	8	82.90	78.25
10	2	9	121.69	100.92
10	2	10	77.58	82.28
14	2	1	286.57	327.72
14	2	2	222.92	233.34
14	2	3	39.01	12.61
14	2	4	109.40	93.56
14	2	5	201.53	189.78
14	2	6	180.10	163.28
14	2	7	147.29	140.56
14	2	8	75.53	66.14
18	2	1	196.43	204.00
18	2	2	281.45	299.09
18	2	3	225.68	226.75
18	2	4	163.05	165.33
18	2	7	104.93	107.07
18	2	8	116.13	105.87
18	2	9	112.08	103.33
18	2	10	56.91	61.41
22	2	1	133.51	140.24
22	2	3	137.79	126.02
22	2	4	166.56	169.83
22	2	5	183.09	183.04
22	2	6	135.30	132.13
22	2	7	81.63	72.82
22	2	10	41.17	51.80
26	2	1	268.02	281.76
26	2	2	299.03	294.47
26	2	3	178.38	170.03
26	2	4	96.50	93.29
26	2	6	64.35	68.93
26	2	7	99.69	108.44
26	2	8	87.41	84.53
26	2	9	67.89	65.55
30	2	2	108.23	99.54
30	2	3	160.42	156.76
30	2	4	181.98	167.54
30	2	5	124.82	122.61
30	2	6	76.71	70.84
30	2	9	49.41	61.92
34	2	1	173.93	193.94
34	2	2	158.83	157.25
34	2	3	50.60	44.90
34	2	5	85.85	89.47
34	2	6	92.44	97.56
34	2	7	86.66	93.29
34	2	8	56.98	58.86
34	2	9	33.66	26.13
38	2	1	105.69	116.64
38	2	2	160.54	181.53

ศูนย์วิทยทรัพยากร
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Table 5.11 (continued)

H	K	L	YOE	YC
38	2	3	165.83	169.69
38	2	4	118.66	149.01
38	2	5	79.69	75.72
38	2	6	33.48	29.73
38	2	8	26.53	42.00
42	2	1	94.37	107.07
42	2	2	52.41	46.36
42	2	4	64.33	75.17
42	2	5	87.82	94.18
42	2	6	83.72	85.91
42	2	7	60.68	54.63
42	2	8	28.53	26.84
46	2	1	94.78	111.33
46	2	2	113.96	134.47
46	2	3	87.88	94.67
46	2	4	64.88	62.73
46	2	7	33.31	52.94
50	2	3	55.10	82.57
50	2	4	76.24	103.04
50	2	5	66.51	84.29
50	2	6	62.11	66.75
50	2	7	24.07	25.51
54	2	1	75.25	94.51
54	2	2	61.97	83.59
54	2	3	45.91	41.68
58	2	3	37.75	60.02
58	2	4	40.53	63.91
58	2	5	29.01	36.53
62	2	1	42.59	50.93
62	2	2	29.25	23.83
-2	2	1	486.32	445.26
-2	2	2	469.05	508.92
-2	2	3	291.84	294.64
-2	2	4	183.74	175.29
-2	2	6	94.35	82.48
-2	2	7	181.90	153.63
-2	2	8	142.95	132.06
-2	2	9	130.20	114.04
-2	2	10	57.99	54.92
-6	2	1	156.21	129.18
-6	2	2	65.57	51.29
-6	2	3	187.54	208.12
-6	2	4	235.58	233.84
-6	2	5	300.41	291.48
-6	2	6	238.65	210.65
-6	2	7	161.43	157.85
-6	2	8	64.19	52.30
-6	2	10	62.74	55.11
-10	2	1	280.05	299.18
-10	2	2	345.43	404.32
-10	2	3	327.39	362.69
-10	2	4	280.20	269.67
-10	2	5	141.92	129.07
-10	2	7	87.40	73.53
-10	2	8	121.95	106.51

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Table 5.11 (continued)

H	K	L	YOE	YC
-10	2	9	112.05	113.73
-10	2	10	87.60	83.34
-14	2	1	350.53	375.07
-14	2	2	285.45	313.71
-14	2	3	63.66	51.23
-14	2	4	57.12	43.38
-14	2	5	174.85	180.54
-14	2	6	187.34	168.75
-14	2	7	197.27	173.41
-14	2	8	111.08	92.70
-14	2	9	51.12	50.07
-18	2	1	95.86	119.11
-18	2	2	194.90	227.54
-18	2	3	238.83	319.42
-18	2	4	283.42	280.57
-18	2	5	227.42	221.82
-18	2	6	133.97	109.14
-18	2	8	56.26	52.72
-18	2	9	93.29	81.52
-18	2	10	87.99	89.37
-22	2	1	282.62	320.81
-22	2	2	288.24	323.04
-22	2	3	181.59	175.40
-22	2	4	120.60	80.25
-22	2	5	69.04	62.39
-22	2	6	108.14	106.10
-22	2	7	165.71	150.12
-22	2	8	130.95	113.31
-22	2	9	101.20	86.94
-26	2	1	119.29	114.69
-26	2	3	140.67	138.17
-26	2	4	180.37	165.65
-26	2	5	212.50	195.54
-26	2	6	136.96	125.97
-26	2	7	85.56	79.40
-26	2	9	50.98	38.58
-30	2	1	181.08	217.30
-30	2	2	249.36	259.10
-30	2	3	235.52	219.61
-30	2	4	179.16	150.87
-30	2	5	64.78	46.08
-30	2	7	97.91	91.37
-30	2	8	118.14	102.94
-30	2	9	109.09	96.52
-34	2	1	152.24	161.15
-34	2	2	109.03	105.59
-34	2	4	97.46	81.33
-34	2	5	176.56	152.00
-34	2	6	152.56	128.36
-34	2	7	138.88	116.06
-34	2	8	62.82	48.22
-6	2	0	513.51	455.05
-38	2	1	58.89	59.74
-38	2	2	117.57	119.54
-38	2	3	184.56	158.07

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Table 5.11 (continued)

H	K	L	YDE	YC
-38	2	4	163.00	133.22
-38	2	5	106.62	88.95
-38	2	8	88.69	73.70
-42	2	1	163.48	159.93
-42	2	2	134.56	140.61
-42	2	3	69.88	56.72
-42	2	5	84.87	78.63
-42	2	6	110.27	96.45
-42	2	7	120.65	111.55
-42	2	8	75.11	76.45
-46	2	2	52.95	49.12
-46	2	3	111.82	114.49
-46	2	4	110.34	119.95
-46	2	5	129.51	118.28
-46	2	6	82.38	68.59
-46	2	7	27.18	29.75
-50	2	1	91.13	97.55
-50	2	2	98.84	106.39
-50	2	3	77.91	78.38
-50	2	6	50.50	55.35
-50	2	7	82.34	81.97
-54	2	1	37.03	53.90
-54	2	3	51.25	46.38
-54	2	4	82.14	73.24
-54	2	5	96.37	97.78
-54	2	6	68.23	79.11
-58	2	1	66.15	67.80
-58	2	2	65.73	89.17
-58	2	3	90.40	95.45
-58	2	4	68.04	73.49
-58	2	5	29.85	40.05
-62	2	1	43.14	58.64
-62	2	2	26.66	38.48
-62	2	4	28.93	30.95

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