

# SDPD\_\_RR1

RR1

Lab diffractometer data, 48 lines, some Pyrochlore impurity (cubic, a = c.9.8A)

**\*\* Summary of Results \*\***

Crystal system: Monoclinic

Unit cell, after correction for specimen displacement (refined):

| a       | b       | c      | alpha | beta  | gamma | Volume   |
|---------|---------|--------|-------|-------|-------|----------|
| 10.3228 | 14.7875 | 8.5290 | 90.00 | 91.35 | 90.00 | 1301.776 |

(see Chekcell's cellref output at the end of this file for esd's, etc.)

Systematic absense condition(s):

h0l: h+l = 2n  
0k0: k = 2n

Spacegroup(s) consistent with this condition:

No. 14 [P2(1)/c], in the setting P2(1)/n

All 33 reliable non-impurity lines indexed out of the 48 reported by PowderX, of which 10 were judged to be due to (or seriously affected by) the declared Pyrochlore impurity. After checking against the original profile a further 5 were also considered too unreliable to be included, as discussed below.

Unindexed line(s):

None

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**\*\* Description of indexing procedure \*\***

RR1 = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002)  
saved by Crysfire 2002 v9.45.25, 12Sep02 at 16:50:51

Self-calibration for dataset RR1 on 12Sep02 at 16:52:48 (v9.45.25)

RR1 = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002)

Seeking Specimen-Displacement correction, max Q-difference = 2.00 QU.

Histogram of specimen-displacement T ratio vs line-pair number

0.0010  
0.0009  
0.0008  
0.0007  
0.0006  
0.0005  
0.0004  
0.0003  
0.0002  
0.0001

0.0000 002  
-0.0001 001 004 005 006  
-0.0002 003  
-0.0003  
-0.0004  
-0.0005  
-0.0006  
-0.0007  
-0.0008  
-0.0009  
-0.0010

List of Possible Specimen-Displacement T ratios found by Self-Calibration

| Trial<br>Pair | 1st<br>Line | 2Theta1<br>deg | 2nd<br>Line | 2Theta2<br>deg | T (=-S/R)   | =Z2Theta<br>at 2Th=0 |
|---------------|-------------|----------------|-------------|----------------|-------------|----------------------|
| 1             | 1           | 11.9500        | 11          | 24.0420        | -0.00007944 | 0.00910              |
| 2             | 2           | 13.2990        | 14          | 26.7820        | -0.00000434 | 0.00050              |
| 3             | 4           | 15.6070        | 23          | 31.5360        | -0.00018382 | 0.02106              |
| 4             | 10          | 23.6290        | 42          | 48.3620        | -0.00012270 | 0.01406              |
| 5             | 11          | 24.0420        | 43          | 49.2420        | -0.00006776 | 0.00776              |
| 6             | 14          | 26.7820        | 47          | 55.2020        | -0.00010954 | 0.01255              |

Mean and esd of listed T ratios = -0.0000946 +- 0.0000601 deg

Whether this is meaningful is a matter for user judgement - several cycles of iteration may be required, adjusting the max Q-difference and perhaps T

Applying specimen-displacement ratio T = -0.0000946 based on mean of list

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RR1SCT = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002), SCT=-0.000095

\*\* ITO12 on RR1SCT: Crysfire 2002 v9.45.25 QDAT-file, 12Sep02 at 17:20:15  
RR1SCT = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002), SCT=-0.000095  
First 40 obs lines (out of 48), wavelength = 1.5406000  
Volmax not required, Z2Theta correction = 0.0 deg  
Special ITO12 parameters: (none)

IT012 post-processor ITLOG v1.2 started on 12Sep02 at 17:20:16

RR1SCT = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002), SCT=-0.000095

4 "most probable" solutions reported (scan 1):

| I20 | Merit | Volume  | V/V1 | BL | IndexProg | Date    | Time     | Pedig | a       | b       | c      | alpha   | beta    | gamma  | Q(A)    | Q(B)   | Q(C)    | Q(D)   | Q(E)   | Q(F)    |
|-----|-------|---------|------|----|-----------|---------|----------|-------|---------|---------|--------|---------|---------|--------|---------|--------|---------|--------|--------|---------|
| 18  | 26.3  | 1302.41 | 1.00 | P  | ITO12/log | 12Sep02 | 17:20:16 | 227   | 10.3202 | 14.7902 | 8.5348 | 90.000  | 91.296  | 90.000 | 93.939  | 45.714 | 137.352 | .000   | 5.140  | .000    |
| 17  | 20.6  | 730.12  | .56  | P  | ITO12/log | 12Sep02 | 17:20:16 | 162   | 8.9573  | 11.0507 | 8.2770 | 110.948 | 107.356 | 85.021 | 136.878 | 93.936 | 182.412 | 91.307 | 90.784 | 5.050   |
| 17  | 8.3   | 1611.25 | 1.24 | P  | ITO12/log | 12Sep02 | 17:20:16 | 233   | 11.5490 | 18.9131 | 7.6515 | 100.603 | 97.927  | 80.748 | 77.935  | 29.507 | 179.026 | 24.065 | 26.380 | -13.339 |
| 17  | 6.5   | 2388.40 | 1.83 | P  | ITO12/log | 12Sep02 | 17:20:16 | 333   | 23.3235 | 13.0202 | 7.8663 | 90.000  | 91.063  | 90.000 | 18.389  | 58.988 | 161.662 | .000   | 2.023  | .000    |

ITLOG run finished on 12Sep02 at 17:20:16 (ITO12 elapsed time not available)

\*\* TREOR90 on RR1SCT: Crysfire 2002 v9.45.25 QDAT-file, 12Sep02 at 17:22:23  
RR1SCT = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002), SCT=-0.000095  
First 25 obs lines (out of 48), wavelength = 1.5406000  
Volmax = 6000.0000 (default), Z2Theta correction = 0.0 deg  
Special TREOR90 parameters: Del2Th = 0.040 (default)

TREOR90 post-processor TRLOG v1.2 started on 12Sep02 at 17:22:26

RR1SCT = Sample 1 - sdpdrr2 - Cu XRD 1.54060 (11-7-2002), SCT=-0.000095

9 solutions reported (up to 25 obs lines, max cell side = 25.0, max volume = 6000.0)

| I20 | Merit | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  | a       | b       | c      | alpha  | beta    | gamma  | Q(A)    | Q(B)     | Q(C)     | Q(D)  | Q(E)     | Q(F)  |
|-----|-------|----------|------|----|-------------|---------|----------|--------|---------|---------|--------|--------|---------|--------|---------|----------|----------|-------|----------|-------|
| 19  | 11    | 1301.829 | 1.00 | P  | TREOR90/log | 12Sep02 | 17:22:26 | Mon__7 | 10.3089 | 14.8023 | 8.5334 | 90.000 | 91.262  | 90.000 | 94.1435 | 45.6397  | 137.3947 | .0000 | 5.0103   | .0000 |
| 18  | 16    | 649.325  | .50  | P  | TREOR90/log | 12Sep02 | 17:22:26 | Mon__2 | 10.3333 | 7.3894  | 8.5066 | 90.000 | 91.457  | 90.000 | 93.7136 | 183.1407 | 138.2830 | .0000 | 5.7878   | .0000 |
| 18  | 16    | 649.325  | .50  | P  | TREOR90/log | 12Sep02 | 17:22:26 | Mon__5 | 13.2163 | 7.3894  | 8.5066 | 90.000 | 128.592 | 90.000 | 93.7137 | 183.1408 | 226.2093 | .0000 | 181.6400 | .0000 |

18 11 1283.367 .99 P TREOR90/log 12Sep02 17:22:26 Mon\_\_1 13.0415 14.7831 6.6809 90.000 94.882 90.000 59.2248 45.7580 225.6756 .0000 19.6782 .0000  
18 10 1300.365 1.00 P TREOR90/log 12Sep02 17:22:26 Mon\_\_4 10.3448 14.7869 8.5038 90.000 91.483 90.000 93.5083 45.7349 138.3765 .0000 5.8873 .0000  
18 8 1302.453 1.00 P TREOR90/log 12Sep02 17:22:26 Mon\_\_3 13.5625 7.3915 13.2345 90.000 100.975 90.000 56.4093 183.0362 59.2407 .0000 22.0101 .0000  
18 7 2435.911 1.87 P TREOR90/log 12Sep02 17:22:26 Ort\_\_2 21.6300 15.2290 7.3949 90.000 90.000 90.000 21.3741 43.1179 182.8658 .0000 .0000 .0000  
17 9 892.114 .69 P TREOR90/log 12Sep02 17:22:26 Mon\_\_6 16.0657 6.1425 9.8166 90.000 112.942 90.000 45.6853 265.0374 122.3628 .0000 58.2879 .0000  
17 7 1599.646 1.23 P TREOR90/log 12Sep02 17:22:26 Ort\_\_1 15.0025 14.4194 7.3945 90.000 90.000 90.000 44.4295 48.0954 182.8845 .0000 .0000 .0000  
(TREOR90 does not report elapsed times)  
TRLOG run finished on 12Sep02 at 17:22:26

| I20 | Merit         | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  | a       | b       | c      | alpha  | beta    | gamma  | Q(A)    | Q(B)     | Q(C)     | Q(D)  | Q(E)     | Q(F)  |
|-----|---------------|----------|------|----|-------------|---------|----------|--------|---------|---------|--------|--------|---------|--------|---------|----------|----------|-------|----------|-------|
| ..  | (soln 28)     |          |      |    |             |         |          |        |         |         |        |        |         |        |         |          |          |       |          |       |
| 19  | 11            | 1301.829 | .83  | P  | TREOR90/log | 12Sep02 | 17:22:26 | Mon__7 | 10.3089 | 14.8023 | 8.5334 | 90.000 | 91.262  | 90.000 | 94.1435 | 45.6397  | 137.3947 | .0000 | 5.0103   | .0000 |
| ..  | (solns 35-37) |          |      |    |             |         |          |        |         |         |        |        |         |        |         |          |          |       |          |       |
| 18  | 26.3          | 1302.41  | .83  | P  | ITO12/log   | 12Sep02 | 17:20:16 | 227    | 10.3202 | 14.7902 | 8.5348 | 90.000 | 91.296  | 90.000 | 93.939  | 45.714   | 137.352  | .000  | 5.140    | .000  |
| 18  | 16            | 649.325  | .41  | P  | TREOR90/log | 12Sep02 | 17:22:26 | Mon__5 | 13.2163 | 7.3894  | 8.5066 | 90.000 | 128.592 | 90.000 | 93.7137 | 183.1408 | 226.2093 | .0000 | 181.6400 | .0000 |
| 18  | 16            | 649.325  | .41  | P  | TREOR90/log | 12Sep02 | 17:22:26 | Mon__2 | 10.3333 | 7.3894  | 8.5066 | 90.000 | 91.457  | 90.000 | 93.7136 | 183.1407 | 138.2830 | .0000 | 5.7878   | .0000 |

This suggests that the correct cell is monoclinic and is either the 1300 A3 cell or its V/2 650 A3 subcell. However, at least 2 Pyrochlore impurity lines are interfering seriously with the correct indexing (in fact there turn out to be around 10, as we shall see later, but these two low-angle lines are the most prominent)

Used the given impurity information (Pyrochlore, cubic, a = c.9.8A, determined here as 9.816A) to remove 2 impurity lines at 2Theta = 15.618 deg (Obs 4, Pyr 111) and 31.546 (Obs 23, Pyr 222)

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RR1-2 = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochlore lines 111,222 saved by Crysfire 2002 v9.45.25, 12Sep02 at 22:26:24

Self-calibration for dataset RR1-2 on 12Sep02 at 22:29:15 (v9.45.25)

RR1-2 = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochlore lines 111,222

Seeking Specimen-Displacement correction, max Q-difference = 2.00 QU.

Histogram of specimen-displacement T ratio vs line-pair number

0.0010  
0.0009  
0.0008  
0.0007  
0.0006  
0.0005  
0.0004  
0.0003  
0.0002  
0.0001  
0.0000 002  
-0.0001 001 003 004 005  
-0.0002  
-0.0003  
-0.0004  
-0.0005  
-0.0006  
-0.0007  
-0.0008  
-0.0009  
-0.0010

List of Possible Specimen-Displacement T ratios found by Self-Calibration

| Trial | 1st  | 2Theta1 | 2nd  | 2Theta2 |           | =Z2Theta |
|-------|------|---------|------|---------|-----------|----------|
| Pair  | Line | deg     | Line | deg     | T (=-S/R) | at 2Th=0 |

|   |    |         |    |         |             |         |
|---|----|---------|----|---------|-------------|---------|
| 1 | 1  | 11.9500 | 10 | 24.0420 | -0.00007944 | 0.00910 |
| 2 | 2  | 13.2990 | 13 | 26.7820 | -0.00000434 | 0.00050 |
| 3 | 9  | 23.6290 | 40 | 48.3620 | -0.00012270 | 0.01406 |
| 4 | 10 | 24.0420 | 41 | 49.2420 | -0.00006776 | 0.00776 |
| 5 | 13 | 26.7820 | 45 | 55.2020 | -0.00010954 | 0.01255 |

Mean and esd of listed T ratios = -0.0000768 +- 0.0000461 deg

Whether this is meaningful is a matter for user judgement - several cycles of iteration may be required, adjusting the max Q-difference and perhaps T

Applying user-specified specimen-displacement ratio T = -0.0000800

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RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.00008  
saved by Crysfire 2002 v9.45.25, 12Sep02 at 22:30:23  
after SC command: T (Z2Th=0, user-specified T = -0.00008000)

| I20 | Merit | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  | a       | b       | c       | alpha   | beta    | gamma  | Q (A)    | Q (B)    | Q (C)    | Q (D)   | Q (E)    | Q (F)    |
|-----|-------|----------|------|----|-------------|---------|----------|--------|---------|---------|---------|---------|---------|--------|----------|----------|----------|---------|----------|----------|
| 20  | 16.1  | 1303.72  | 1.00 | P  | ITO12/log   | 12Sep02 | 22:31:22 | 227    | 10.3207 | 14.8024 | 8.5360  | 90.000  | 91.294  | 90.000 | 93.930   | 45.639   | 137.313  | .000    | 5.129    | .000     |
| 19  | 14.45 | 1302.384 | 1.00 | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 2      | 10.3162 | 14.7987 | 8.5331  | 90.000  | 91.289  | 90.000 | 94.0117  | 45.6615  | 137.4072 | .0000   | 5.1125   | .0000    |
| 19  | 13.53 | 816.406  | .63  | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 16     | 8.3313  | 13.3344 | 7.4610  | 90.070  | 97.600  | 96.366 | 148.5001 | 56.9560  | 182.8874 | 3.2924  | 43.9092  | 20.6057  |
| 19  | 11    | 649.580  | .50  | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon_5  | 13.2256 | 7.3837  | 8.5183  | 90.000  | 128.658 | 90.000 | 93.7537  | 183.4224 | 226.0028 | .0000   | 181.8575 | .0000    |
| 18  | 21    | 1301.282 | 1.00 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__2 | 13.5426 | 7.3937  | 13.2349 | 90.000  | 100.904 | 90.000 | 56.5482  | 182.9266 | 59.2088  | .0000   | 21.8911  | .0000    |
| 18  | 12.69 | 1953.245 | 1.50 | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 3      | 10.3173 | 22.1876 | 8.5348  | 90.000  | 91.297  | 90.000 | 93.9914  | 20.3133  | 137.3535 | .0000   | 5.1417   | .0000    |
| 18  | 12    | 649.401  | .50  | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__4 | 10.3358 | 7.3918  | 8.5032  | 90.000  | 91.587  | 90.000 | 93.6796  | 183.0185 | 138.4100 | .0000   | 6.3066   | .0000    |
| 18  | 12    | 649.401  | .50  | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__1 | 10.3358 | 7.3918  | 8.5032  | 90.000  | 91.587  | 90.000 | 93.6796  | 183.0185 | 138.4100 | .0000   | 6.3066   | .0000    |
| 18  | 11.66 | 769.930  | .59  | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 7      | 10.0114 | 11.5545 | 7.0708  | 105.064 | 102.857 | 87.517 | 104.9949 | 80.3475  | 225.3062 | 69.1385 | 67.3591  | 2.8312   |
| 18  | 9.86  | 971.516  | .75  | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 14     | 9.7325  | 15.1543 | 6.7592  | 100.185 | 93.414  | 82.218 | 107.6874 | 45.6876  | 226.2478 | 34.6968 | 11.3992  | -17.8290 |
| 18  | 6     | 3368.882 | 2.58 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Ort__2 | 19.3845 | 15.1592 | 11.4645 | 90.000  | 90.000  | 90.000 | 26.6128  | 43.5158  | 76.0834  | .0000   | .0000    | .0000    |
| 18  | 4.8   | 3454.03  | 2.65 | P  | ITO12/log   | 12Sep02 | 22:31:22 | 119    | 23.8442 | 8.0845  | 18.3103 | 90.000  | 101.881 | 90.000 | 18.367   | 153.001  | 31.147   | .000    | 9.849    | .000     |
| 17  | 12.3  | 1080.13  | .83  | P  | ITO12/log   | 12Sep02 | 22:31:22 | 164    | 10.5178 | 14.2672 | 8.1107  | 107.163 | 103.694 | 69.991 | 104.780  | 58.880   | 170.410  | 46.978  | 40.415   | -46.081  |
| 17  | 10.90 | 966.522  | .74  | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 12     | 11.6646 | 12.3124 | 7.5935  | 99.942  | 100.261 | 64.967 | 90.7836  | 81.3204  | 181.2672 | 26.4943 | 30.2072  | -69.5707 |
| 17  | 10.9  | 1072.94  | .82  | P  | ITO12/log   | 12Sep02 | 22:31:22 | 121    | 10.4268 | 13.4777 | 7.9144  | 103.264 | 93.337  | 95.903 | 93.633   | 58.953   | 169.731  | 47.430  | 21.303   | 17.770   |
| 17  | 8     | 2751.493 | 2.11 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Ort__1 | 15.0409 | 14.2094 | 12.8741 | 90.000  | 90.000  | 90.000 | 44.2031  | 49.5276  | 60.3342  | .0000   | .0000    | .0000    |
| 17  | 7     | 1112.407 | .85  | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__3 | 16.0539 | 8.4376  | 8.5731  | 90.000  | 106.683 | 90.000 | 42.2857  | 140.4619 | 148.2772 | .0000   | 45.4639  | .0000    |
| 16  | 41.30 | 650.903  | .50  | P  | KOHLv7.01b  | 12Sep02 | 22:46:11 | 1      | 10.3184 | 7.3940  | 8.5336  | 90.000  | 91.292  | 90.000 | 93.9707  | 182.9117 | 137.3896 | .0000   | 5.1243   | .0000    |

\*\* ITO12 on RR1-2SCT: Crysfire 2002 v9.45.25 QDAT-file, 12Sep02 at 22:31:19  
RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000  
First 40 obs lines (out of 46), wavelength = 1.5406000  
Volmax not required, Z2Theta correction = 0.0 deg  
Special ITO12 parameters: (none)

ITO12 post-processor ITLOG v1.2 started on 12Sep02 at 22:31:22  
RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000  
4 "most probable" solutions reported (scan 1):  
I20 Merit Volume V/V1 BL IndexProg Date Time Pedig a b c alpha beta gamma Q (A) Q (B) Q (C) Q (D) Q (E) Q (F)  
20 16.1 1303.72 1.00 P ITO12/log 12Sep02 22:31:22 227 10.3207 14.8024 8.5360 90.000 91.294 90.000 93.930 45.639 137.313 .000 5.129 .000  
17 12.3 1080.13 .83 P ITO12/log 12Sep02 22:31:22 164 10.5178 14.2672 8.1107 107.163 103.694 69.991 104.780 58.880 170.410 46.978 40.415 -46.081  
17 10.9 1072.94 .82 P ITO12/log 12Sep02 22:31:22 121 10.4268 13.4777 7.9144 103.264 93.337 95.903 93.633 58.953 169.731 47.430 21.303 17.770  
18 4.8 3454.03 2.65 P ITO12/log 12Sep02 22:31:22 119 23.8442 8.0845 18.3103 90.000 101.881 90.000 18.367 153.001 31.147 .000 9.849 .000  
ITLOG run finished on 12Sep02 at 22:31:22 (ITO12 elapsed time not available)

\*\* TREOR90 on RR1-2SCT: Crysfire 2002 v9.45.25 QDAT-file, 12Sep02 at 22:35:59  
RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000  
First 25 obs lines (out of 46), wavelength = 1.5406000  
Volmax = 6000.0000 (default), Z2Theta correction = 0.0 deg  
Special TREOR90 parameters: Del2Th = 0.040 (default)

TREOR90 post-processor TRLOG v1.2 started on 12Sep02 at 22:36:05  
RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000

7 solutions reported (up to 25 obs lines, max cell side = 25.0, max volume = 6000.0)

| I20 | Merit | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  | a       | b       | c       | alpha  | beta    | gamma  | Q(A)    | Q(B)     | Q(C)     | Q(D)  | Q(E)     | Q(F)  |
|-----|-------|----------|------|----|-------------|---------|----------|--------|---------|---------|---------|--------|---------|--------|---------|----------|----------|-------|----------|-------|
| 19  | 11    | 649.580  | 1.00 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__5 | 13.2256 | 7.3837  | 8.5183  | 90.000 | 128.658 | 90.000 | 93.7537 | 183.4224 | 226.0028 | .0000 | 181.8575 | .0000 |
| 18  | 21    | 1301.282 | 2.00 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__2 | 13.5426 | 7.3937  | 13.2349 | 90.000 | 100.904 | 90.000 | 56.5482 | 182.9266 | 59.2088  | .0000 | 21.8911  | .0000 |
| 18  | 12    | 649.401  | 1.00 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__1 | 10.3358 | 7.3918  | 8.5032  | 90.000 | 91.587  | 90.000 | 93.6796 | 183.0185 | 138.4100 | .0000 | 6.3066   | .0000 |
| 18  | 12    | 649.401  | 1.00 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__4 | 10.3358 | 7.3918  | 8.5032  | 90.000 | 91.587  | 90.000 | 93.6796 | 183.0185 | 138.4100 | .0000 | 6.3066   | .0000 |
| 18  | 6     | 3368.882 | 5.19 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Ort__2 | 19.3845 | 15.1592 | 11.4645 | 90.000 | 90.000  | 90.000 | 26.6128 | 43.5158  | 76.0834  | .0000 | .0000    | .0000 |
| 17  | 8     | 2751.493 | 4.24 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Ort__1 | 15.0409 | 14.2094 | 12.8741 | 90.000 | 90.000  | 90.000 | 44.2031 | 49.5276  | 60.3342  | .0000 | .0000    | .0000 |
| 17  | 7     | 1112.407 | 1.71 | P  | TREOR90/log | 12Sep02 | 22:36:05 | Mon__3 | 16.0539 | 8.4376  | 8.5731  | 90.000 | 106.683 | 90.000 | 42.2857 | 140.4619 | 148.2772 | .0000 | 45.4639  | .0000 |

(TREOR90 does not report elapsed times)

TRLOG run finished on 12Sep02 at 22:36:05

\*\* FJZN6 on RR1-2SCT: Crysfire 2002 v9.45.25 QDAT-file, 12Sep02 at 22:40:21

RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000

First 40 obs lines (out of 46), wavelength = 1.5406000

Volmax not required, Z2Theta correction = 0.0 deg

Special FJZN6 parameters: (none)

FJZN v6.22a run started on 12Sep02 at 22:40:29

RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000

\*\* No solutions found with I20>15 and Merit > 7.0 \*\*

FJZN v6.22a run finished on 12Sep02 at 22:40:32 (Elapsed time: 3.25 sec)

\*\* KOHL on RR1-2SCT: Crysfire 2002 v9.45.25 QDAT-file, 12Sep02 at 22:41:12

RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000

First 30 obs lines (out of 46), wavelength = 1.5406000

Volmax not required, Z2Theta correction = 0.0 deg

Special KOHL parameters: Nspuri = 0 (default), IORT = OMT (all three, default)

KOHL v7.01b run started on 12Sep02 at 22:41:21

RR1-2SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyrochl 111,222, SCT=-0.000

7 solutions found with I20>15 and Merit > 9.0

| I20 | Merit | Volume   | V/V1 | BL | IndexProg  | Date    | Time     | Pedig | a       | b       | c      | alpha   | beta    | gamma  | Q(A)     | Q(B)     | Q(C)     | Q(D)    | Q(E)    | Q(F)     |
|-----|-------|----------|------|----|------------|---------|----------|-------|---------|---------|--------|---------|---------|--------|----------|----------|----------|---------|---------|----------|
| 19  | 14.45 | 1302.384 | 1.00 | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 2     | 10.3162 | 14.7987 | 8.5331 | 90.000  | 91.289  | 90.000 | 94.0117  | 45.6615  | 137.4072 | .0000   | 5.1125  | .0000    |
| 19  | 13.53 | 816.406  | .63  | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 16    | 8.3313  | 13.3344 | 7.4610 | 90.070  | 97.600  | 96.366 | 148.5001 | 56.9560  | 182.8874 | 3.2924  | 43.9092 | 20.6057  |
| 18  | 12.69 | 1953.245 | 1.50 | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 3     | 10.3173 | 22.1876 | 8.5348 | 90.000  | 91.297  | 90.000 | 93.9914  | 20.3133  | 137.3535 | .0000   | 5.1417  | .0000    |
| 18  | 11.66 | 769.930  | .59  | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 7     | 10.0114 | 11.5545 | 7.0708 | 105.064 | 102.857 | 87.517 | 104.9949 | 80.3475  | 225.3062 | 69.1385 | 67.3591 | 2.8312   |
| 18  | 9.86  | 971.516  | .75  | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 14    | 9.7325  | 15.1543 | 6.7592 | 100.185 | 93.414  | 82.218 | 107.6874 | 45.6876  | 226.2478 | 34.6968 | 11.3992 | -17.8290 |
| 17  | 10.90 | 966.522  | .74  | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 12    | 11.6646 | 12.3124 | 7.5935 | 99.942  | 100.261 | 64.967 | 90.7836  | 81.3204  | 181.2672 | 26.4943 | 30.2072 | -69.5707 |
| 16  | 41.30 | 650.903  | .50  | P  | KOHLv7.01b | 12Sep02 | 22:46:11 | 1     | 10.3184 | 7.3940  | 8.5336 | 90.000  | 91.292  | 90.000 | 93.9707  | 182.9117 | 137.3896 | .0000   | 5.1243  | .0000    |

KOHL v7.01b run finished on 12Sep02 at 22:46:11 (Elapsed time: 289.46 sec)

These results confirm the 1302 A3 monoclinic P cell with the 650 A3 subcell as another contender. The 1953 A3 1.5V supercell also reported by is less likely.

The Pyrochlore 311 line was then also removed, to try to clarify the situation.

-----

RR1-3SCT = Sample 1 sdpdrr2, CuKa (11-7-2002) w/o Pyr 111,311,222, SCT=-0.00008

saved by Crysfire 2002 v9.45.25, 12Sep02 at 22:56:39

after ED command: 45 obs lines after edit (previously 46 lines)

< Various further output omitted >

It had become sufficiently clear from the Crysfire results that this 1302 A3 family of primitive monoclinic cells contained the physically correct lattice.

Returned at this point to the original un-calibrated PowderX 2Theta data, to filter all 10 lines that were either Pyrochlore lines or too close to such lines to be reliable, leaving 38 non-impurity lines in the RR1-P dataset.

This dataset was then used to re-estimate the specimen-displacement correction by self-calibration:

Self-calibration for dataset RR1-P on 10Oct02 at 13:08:21 (v9.45.26)

RR1-P = Sample 1 (sdpdr2) CuXRD 1.54060 (11-7-2002) -10Pyr(38obs)

Seeking Specimen-Displacement correction, max Q-difference = 1.50 QU.

Histogram of specimen-displacement T ratio vs line-pair number

0.0010  
0.0009  
0.0008  
0.0007  
0.0006  
0.0005  
0.0004  
0.0003  
0.0002  
0.0001  
0.0000 002  
-0.0001 001 003  
-0.0002  
-0.0003  
-0.0004  
-0.0005  
-0.0006  
-0.0007  
-0.0008  
-0.0009  
-0.0010

List of Possible Specimen-Displacement T ratios found by Self-Calibration

| Trial<br>Pair | 1st<br>Line | 2Theta1<br>deg | 2nd<br>Line | 2Theta2<br>deg | T (=-S/R)   | =Z2Theta<br>at 2Th=0 |
|---------------|-------------|----------------|-------------|----------------|-------------|----------------------|
| 1             | 1           | 11.9500        | 10          | 24.0420        | -0.00007944 | 0.00910              |
| 2             | 2           | 13.2990        | 12          | 26.7820        | -0.00000434 | 0.00050              |
| 3             | 10          | 24.0420        | 35          | 49.2420        | -0.00006776 | 0.00776              |

Mean and esd of listed T ratios = -0.0000505 +- 0.0000404 deg

Whether this is meaningful is a matter for user judgement - several cycles of iteration may be required, adjusting the max Q-difference and perhaps T

Applying user-specified specimen-displacement ratio T = -0.0000500

List Observed Pattern for dataset RR1-P on 10Oct02 at 13:08:54 (v9.45.26)

RR1-P = Sample 1 (sdpdr2) CuXRD 1.54060 (11-7-2002) -10Pyr(38obs)

Wavelength = 1.540600 Angstroms  
After self-calibration: T (Z2Th=0, user-specified T = -0.00005000)  
No zero correction applied

| Obs<br>No. | Corr'd<br>2Theta | Corr'd<br>Q(Obs) | d(obs)   | Qualitative<br>Intensity | Quantitative<br>Intensity |
|------------|------------------|------------------|----------|--------------------------|---------------------------|
| 1          | 11.9557          | 182.78833        | 7.396492 | m                        | 5882.200                  |
| 2          | 13.3047          | 226.16852        | 6.649422 | vs                       | 31622.300                 |
| 3          | 13.6157          | 236.81513        | 6.498233 | vs                       | 27061.200                 |
| 4          | 17.1697          | 375.53134        | 5.160323 | vs                       | 29782.700                 |
| 5          | 17.9207          | 408.82697        | 4.945728 | m                        | 4026.200                  |
| 6          | 18.1547          | 419.48310        | 4.882506 | mw                       | 3382.300                  |

|    |         |            |          |     |           |
|----|---------|------------|----------|-----|-----------|
| 7  | 20.8146 | 549.95981  | 4.264170 | vs  | 25935.100 |
| 8  | 23.2656 | 685.21722  | 3.820198 | mw  | 3422.400  |
| 9  | 23.6346 | 706.81343  | 3.761383 | vvw | 492.000   |
| 10 | 24.0476 | 731.36449  | 3.697712 | ms  | 8390.800  |
| 11 | 25.4366 | 816.86049  | 3.498856 | vvw | 595.000   |
| 12 | 26.7876 | 904.30800  | 3.325384 | m   | 4844.400  |
| 13 | 27.5876 | 958.06497  | 3.230744 | s   | 18418.900 |
| 14 | 27.7276 | 967.62243  | 3.214749 | vs  | 19890.500 |
| 15 | 28.1746 | 998.43584  | 3.164754 | m   | 5776.400  |
| 16 | 29.4355 | 1087.79172 | 3.031986 | mw  | 3204.300  |
| 17 | 29.7025 | 1107.16911 | 3.005336 | m   | 5410.300  |
| 18 | 30.6995 | 1180.92730 | 2.909969 | vw  | 986.800   |
| 19 | 31.7495 | 1260.97781 | 2.816088 | vvw | 715.100   |
| 20 | 34.7015 | 1498.83652 | 2.582991 | m   | 5810.500  |
| 21 | 35.1355 | 1535.37220 | 2.552073 | mw  | 2588.300  |
| 22 | 36.8094 | 1679.98009 | 2.439765 | w   | 2041.200  |
| 23 | 37.0094 | 1697.64457 | 2.427038 | ms  | 10876.900 |
| 24 | 37.3494 | 1727.86224 | 2.405722 | mw  | 3074.300  |
| 25 | 40.3544 | 2005.06462 | 2.233242 | w   | 1679.400  |
| 26 | 41.1354 | 2080.03262 | 2.192628 | mw  | 3066.700  |
| 27 | 42.3053 | 2194.54259 | 2.134656 | mw  | 2608.900  |
| 28 | 42.5103 | 2214.87521 | 2.124836 | vw  | 891.400   |
| 29 | 43.1973 | 2283.59249 | 2.092622 | mw  | 2669.700  |
| 30 | 43.3753 | 2301.54141 | 2.084446 | m   | 4000.500  |
| 31 | 43.3933 | 2303.35977 | 2.083623 | m   | 4356.400  |
| 32 | 45.6073 | 2531.56946 | 1.987491 | mw  | 3364.400  |
| 33 | 47.1243 | 2693.03741 | 1.926987 | vw  | 1169.100  |
| 34 | 48.3672 | 2828.34176 | 1.880330 | vw  | 1211.100  |
| 35 | 49.2472 | 2925.72906 | 1.848770 | m   | 4782.700  |
| 36 | 51.2862 | 3156.32668 | 1.779955 | m   | 5233.200  |
| 37 | 54.2361 | 3501.68529 | 1.689902 | vw  | 1341.900  |
| 38 | 55.5221 | 3656.37832 | 1.653767 | w   | 1746.400  |

This 38-line dataset with T=-0.0005 applied was saved as RR1-PSCT

-----

RR1-PSCT = Sample 1 (sdpdr2) CuXRD 1.54060 -10Pyr (38 obs) SCT=-0.00005

| I20 | Merit  | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  | a       | b       | c       | alpha   | beta    | gamma  | Q (A)    | Q (B)    | Q (C)    | Q (D)  | Q (E)   | Q (F)    |
|-----|--------|----------|------|----|-------------|---------|----------|--------|---------|---------|---------|---------|---------|--------|----------|----------|----------|--------|---------|----------|
| 20  | 18.17  | 1951.782 | 1.00 | P  | KOHLv7.01b  | 10Oct02 | 13:23:35 | 1      | 10.3269 | 22.1812 | 8.5231  | 90.000  | 91.355  | 90.000 | 93.8221  | 20.3251  | 137.7348 | .0000  | 5.3745  | .0000    |
| 20  | 16.60  | 1302.471 | .67  | P  | KOHLv7.01b  | 10Oct02 | 13:23:35 | 3      | 13.5369 | 7.3974  | 13.2431 | 90.000  | 100.838 | 90.000 | 56.5711  | 182.7458 | 59.1090  | .0000  | 21.7459 | .0000    |
| 20  | 11     | 1300.689 | .67  | P  | TREOR90/log | 10Oct02 | 13:20:27 | Mon__1 | 13.5518 | 7.3913  | 13.2283 | 90.000  | 100.995 | 90.000 | 56.5067  | 183.0459 | 59.3040  | .0000  | 22.0810 | .0000    |
| 19  | 70.32  | 650.902  | .33  | P  | KOHLv7.01b  | 10Oct02 | 13:23:35 | 4      | 10.3223 | 7.3947  | 8.5298  | 90.000  | 91.317  | 90.000 | 93.9027  | 182.8785 | 137.5171 | .0000  | 5.2243  | .0000    |
| 19  | 38.16  | 1301.775 | .67  | P  | KOHLv7.01b  | 10Oct02 | 13:23:35 | 2      | 10.3223 | 14.7889 | 8.5297  | 90.000  | 91.317  | 90.000 | 93.9018  | 45.7221  | 137.5175 | .0000  | 5.2231  | .0000    |
| 19  | 6      | 2838.933 | 1.45 | P  | TREOR90/log | 10Oct02 | 13:20:27 | Ort__5 | 14.8131 | 14.4083 | 13.3014 | 90.000  | 90.000  | 90.000 | 45.5730  | 48.1700  | 56.5204  | .0000  | .0000   | .0000    |
| 19  | 5.0    | 2771.25  | 1.42 | P  | IT012/log   | 10Oct02 | 13:12:17 | 668    | 25.1444 | 6.6427  | 16.7585 | 90.000  | 98.091  | 90.000 | 16.136   | 226.626  | 36.326   | .000   | 6.815   | .000     |
| 18  | 12.5   | 699.14   | .36  | P  | IT012/log   | 10Oct02 | 13:12:17 | 354    | 7.1080  | 15.2932 | 6.7049  | 103.035 | 97.341  | 81.659 | 204.167  | 45.710   | 236.665  | 43.883 | 43.352  | -23.244  |
| 18  | 7.3    | 1029.03  | .53  | P  | IT012/log   | 10Oct02 | 13:12:17 | 173    | 10.5399 | 13.2639 | 7.5104  | 90.911  | 90.682  | 78.590 | 93.691   | 59.167   | 177.345  | 2.830  | 2.303   | -29.435  |
| 18  | 6      | 1614.202 | .83  | P  | TREOR90/log | 10Oct02 | 13:20:27 | Ort__3 | 15.1871 | 14.7895 | 7.1867  | 90.000  | 90.000  | 90.000 | 43.3563  | 45.7189  | 193.6138 | .0000  | .0000   | .0000    |
| 18  | 4.3    | 1538.03  | .79  | P  | IT012/log   | 10Oct02 | 13:12:17 | 574    | 10.3501 | 15.1219 | 10.0775 | 102.279 | 92.691  | 86.993 | 93.733   | 45.889   | 103.271  | 29.013 | 7.218   | -5.707   |
| 17  | 13.6   | 815.78   | .42  | P  | IT012/log   | 10Oct02 | 13:12:17 | 337    | 15.2872 | 7.1964  | 7.4155  | 90.000  | 90.396  | 90.000 | 42.792   | 193.094  | 181.861  | .000   | 1.220   | .000     |
| 17  | 11.8   | 609.30   | .31  | P  | IT012/log   | 10Oct02 | 13:12:17 | 351    | 9.5444  | 9.7967  | 6.6554  | 94.241  | 100.795 | 91.014 | 113.884  | 104.875  | 235.427  | 24.725 | 61.942  | 7.040    |
| 17  | 9.10   | 1138.953 | .58  | P  | FJZNV6.22a  | 10Oct02 | 13:16:29 | 121    | 10.2316 | 15.3131 | 7.6188  | 92.914  | 104.000 | 79.540 | 104.6549 | 44.1012  | 182.9980 | 1.3032 | 65.5766 | -23.7306 |
| 17  | 8      | 1603.721 | .82  | P  | TREOR90/log | 10Oct02 | 13:20:27 | Ort__1 | 15.0218 | 14.8326 | 7.1976  | 90.000  | 90.000  | 90.000 | 44.3154  | 45.4531  | 193.0298 | .0000  | .0000   | .0000    |
| 17  | 6      | 5499.749 | 2.82 | P  | TREOR90/log | 10Oct02 | 13:20:27 | Tet__2 | 18.8136 | 18.8136 | 15.5382 | 90.000  | 90.000  | 90.000 | 28.2526  | 28.2526  | 41.4188  | .0000  | .0000   | .0000    |
| 17  | 6      | 4234.221 | 2.17 | P  | TREOR90/log | 10Oct02 | 13:20:27 | Tet__1 | 16.4302 | 16.4302 | 15.6851 | 90.000  | 90.000  | 90.000 | 37.0437  | 37.0437  | 40.6466  | .0000  | .0000   | .0000    |
| 17  | 5.0    | 1606.93  | .82  | P  | IT012/log   | 10Oct02 | 13:12:17 | 341    | 8.7178  | 26.0861 | 7.9110  | 96.204  | 115.779 | 90.735 | 162.998  | 14.936   | 200.246  | 13.805 | 158.584 | 6.594    |
| 16  | 157.28 | 325.475  | .17  | P  | FJZNV6.22a  | 10Oct02 | 13:16:29 | 551    | 6.7701  | 7.3949  | 6.6197  | 90.000  | 100.858 | 90.000 | 226.2072 | 182.8694 | 236.5968 | .0000  | 87.1575 | .0000    |
| 16  | 15.7   | 431.71   | .22  | P  | IT012/log   | 10Oct02 | 13:12:17 | 352    | 6.7945  | 9.8346  | 6.6570  | 95.825  | 101.541 | 83.623 | 227.620  | 105.382  | 236.632  | 25.709 | 88.639  | -28.845  |
| 13  | 10     | 1590.739 | .82  | P  | TREOR90/log | 10Oct02 | 13:20:27 | Ort__2 | 14.8737 | 14.8737 | 7.1906  | 90.000  | 90.000  | 90.000 | 45.2026  | 45.2026  | 193.4082 | .0000  | .0000   | .0000    |
| 10  | 8      | 1609.506 | .82  | P  | TREOR90/log | 10Oct02 | 13:20:27 | Ort__4 | 14.9908 | 14.9908 | 7.1622  | 90.000  | 90.000  | 90.000 | 44.4991  | 44.4991  | 194.9452 | .0000  | .0000   | .0000    |

We're now starting to see some reassuringly high figures of merit, notably M16=157 for the

325.5 A3 V/4 subcell found by FJZN, but also M19=70 for the 651 A3 V/2 subcell and M19=38 for the 1302 A3 1V cell, both found by KOHL. The M20=18 1.5V supercell (if that's what it is) also found by KOHL remains a possible contender, though my money is on the 1V cell (in one of the two non-equivalent versions shown here, with and without B doubled).

It's now time to transfer to Chekcell and see whether spacegroup extinctions can settle these issues, and re-assess PowderX's 2Theta positions vs the original profile:

RR1-PSCT - Chekcell (Best Group)

Soln 4 V/2 (650.9 A3), KOHL M19=70.3

Best Group (Opt Tol = 0.077deg)

| Sp. Group | Sigma   | Indexed | Calculated |
|-----------|---------|---------|------------|
| P2        | 0.02248 | 38      | 180        |
| P21       | 0.02247 | 37      | 178        |
| C2        | 0.02218 | 23      | 89         |
| PM        | 0.02248 | 38      | 180        |
| PC        | 0.02145 | 34      | 154        |
| PA        | 0.02145 | 34      | 154        |
| CM        | 0.02218 | 23      | 89         |
| CC        | 0.02218 | 23      | 77         |
| CA        | 0.02218 | 23      | 77         |
| P2/M      | 0.02248 | 38      | 180        |
| P21/M     | 0.02247 | 37      | 178        |
| C2/M      | 0.02218 | 23      | 89         |
| P2/C      | 0.02145 | 34      | 154        |
| P21/A     | 0.02145 | 33      | 152        |
| P21/C     | 0.02145 | 33      | 152        |
| P21/N     | 0.02247 | 37      | 154        |
| C2/C      | 0.02218 | 23      | 77         |

Best estimated group(s):

|      |         |    |     |
|------|---------|----|-----|
| P2   | 0.02248 | 38 | 180 |
| PM   | 0.02248 | 38 | 180 |
| P2/M | 0.02248 | 38 | 180 |

But see:

|       |         |    |     |
|-------|---------|----|-----|
| P21/N | 0.02247 | 37 | 154 |
|-------|---------|----|-----|

If b were doubled then all 38 lines could be indexed with the 1302 A3 variant that is based on this V/2 subcell, so this is looking the best contender, since it will index all lines while giving a crystallographically-plausible spacegroup.

Re-assessing the 2Thetas vs the profile showed that a further 5 of PowderX's line positions were suspect (obs line numbering based on the re-calibrated 38-line pattern as listed above):

Obs no. 2Theta Reason for exclusion as unreliable

|    |        |                                                    |
|----|--------|----------------------------------------------------|
| 19 | 31.750 | Indistinct shoulder                                |
| 25 | 40.354 | Excessively weak and noisy                         |
| 28 | 42.510 | Indistinct shoulder                                |
| 29 | 43.197 | Indistinct shoulder                                |
| 34 | 48.367 | Weak shoulder on Pyr line 10 (511 & 333) at 48.127 |

These were deleted and the resulting (un-recalibrated) 33-line pattern of hopefully the most reliable RR1 ilnes saved as RR1-X

RR1-X (33 lines after a further 5 deleted)

-----



RR1-X = Sample 1 (sdpdr2) CuXRD 1.54060 (11-7-2002) -15Pyr/x(33obs)

(i.e. The un-recalibrated 33-line pattern with the above 5 lines deleted)

This was loaded into Chekcell and confirmed with Best Group to give primitive monoclinic spacegroup P2(1)/n for the 1302 A3 cell with b-doubled to 14.8A, as in KOHL's cell 4 above (which then had M19=38).

This was submitted for cell refinement of all monoclinic parameters plus the specimen-displacement correction (as defined by Chekcell - differently from Crysfire's self-calibration function), giving the following result:

Chekcell - cell refinement

-----  
RR1-X.CDT  
-----

Number of reflections : 33  
Refinement constraints : NO

Initial values : (Refinement flags on 2nd line)  
e/R(ø) Lambda a b c alpha beta gamma  
.00000 1.54060 10.3220 14.7890 8.5300 90.00 91.32 90.00  
1. 0. 1. 1. 1. 0. 1. 0.

Volume (A\*\*3) : 1301.776

Final values : (Standard errors on 2nd line)  
e/R(ø) Lambda a b c alpha beta gamma  
-.00321 1.54060 10.3228 14.7875 8.5290 90.00 91.35 90.00  
.00064 .00000 .0005 .0007 .0004 .00 .00 .00

Volume (A\*\*3) : 1301.568  
Standard error : .2681

Number of cycles: 3

| H  | K | L | 2Th(obs) | 2Th-Zero | 2Th(Calc) | diff. |
|----|---|---|----------|----------|-----------|-------|
| 0  | 2 | 0 | 11.950   | 11.953   | 11.960    | -.007 |
| -1 | 0 | 1 | 13.299   | 13.302   | 13.302    | .000  |
| 1  | 0 | 1 | 13.610   | 13.613   | 13.616    | -.003 |
| 2  | 0 | 0 | 17.164   | 17.167   | 17.171    | -.004 |
| -1 | 2 | 1 | 17.915   | 17.918   | 17.924    | -.006 |
| 1  | 2 | 1 | 18.149   | 18.152   | 18.160    | -.008 |
| 0  | 0 | 2 | 20.809   | 20.812   | 20.819    | -.007 |
| -2 | 2 | 1 | 23.260   | 23.263   | 23.267    | -.004 |
| 2  | 2 | 1 | 23.629   | 23.632   | 23.634    | -.002 |
| 0  | 4 | 0 | 24.042   | 24.045   | 24.053    | -.008 |
| -1 | 2 | 2 | 25.431   | 25.434   | 25.427    | .007  |
| -2 | 0 | 2 | 26.782   | 26.785   | 26.787    | -.002 |
| -1 | 4 | 1 | 27.582   | 27.585   | 27.583    | .002  |
| -3 | 0 | 1 | 27.722   | 27.725   | 27.711    | .014  |
| 3  | 0 | 1 | 28.169   | 28.172   | 28.179    | -.007 |
| -2 | 2 | 2 | 29.430   | 29.433   | 29.427    | .006  |
| -2 | 4 | 0 | 29.697   | 29.700   | 29.704    | -.004 |
| 3  | 2 | 1 | 30.694   | 30.697   | 30.709    | -.012 |
| -1 | 2 | 3 | 34.696   | 34.699   | 34.699    | .000  |
| 1  | 2 | 3 | 35.130   | 35.133   | 35.082    | .051  |
| 2  | 4 | 2 | 36.804   | 36.807   | 36.798    | .009  |
| -3 | 4 | 1 | 37.004   | 37.007   | 37.016    | -.009 |
| -1 | 3 | 3 | 37.344   | 37.347   | 37.343    | .004  |
| 1  | 4 | 3 | 41.130   | 41.133   | 41.131    | .002  |
| -4 | 2 | 2 | 42.300   | 42.303   | 42.284    | .019  |
| 1  | 6 | 2 | 43.370   | 43.373   | 43.371    | .002  |
| 1  | 6 | 2 | 43.388   | 43.391   | 43.371    | .020  |

|    |   |   |        |        |        |       |
|----|---|---|--------|--------|--------|-------|
| -5 | 2 | 0 | 45.602 | 45.605 | 45.595 | .009  |
| 5  | 2 | 1 | 47.119 | 47.122 | 47.140 | -.018 |
| 0  | 8 | 0 | 49.242 | 49.245 | 49.257 | -.012 |
| 3  | 1 | 4 | 51.281 | 51.284 | 51.283 | .000  |
| -1 | 0 | 5 | 54.231 | 54.234 | 54.270 | -.036 |
| -4 | 6 | 2 | 55.517 | 55.520 | 55.518 | .002  |

Sqrt(Sum(Th O-C)\*\*2)/(Nref-Npar) = .002802

Sqrt(Sum(2Th O-C)\*\*2)/(Nref-Npar) = .001401

\*\* End of description of indexing procedure \*\*

# SDPD\_RR2

RR2 = Sample 2 - sdpdrr2 - Wavelength 0.79764 (23-11-1998)

Synchrotron data, capillary sample

\*\* Summary of Results \*\*

Crystal system: Monoclinic

Unit cell (from KOHL, M20 = 95.4, no 2Theta zero correction thought necessary):

| a       | b      | c       | alpha  | beta    | gamma  | Volume   |
|---------|--------|---------|--------|---------|--------|----------|
| 19.8778 | 8.1933 | 11.2407 | 90.000 | 106.056 | 90.000 | 1759.294 |

Systematic absences condition(s):

h0l: h+l = 2n  
 0k0: k = 2n

Spacegroup(s) consistent with this condition:

No. 14 [P2(1)/c], in the setting P2(1)/n

All 150 lines indexed out of first 150 observed (from 327 reported by PowderX)  
 using 2Theta tolerance 0.019 deg

Unindexed line(s):

None

\*\*\*\*\*

\*\* Outline of indexing procedure \*\*

Crysfire 2002 v9.45.25 (16Sep02)

[Self-calibration (Z2Th): not used for this initial run, and then judged to be unnecessary]

Initial indexing runs gave the following summary file (edited to remove irrelevant solutions):

| I20 | Merit | Volume   | V/V1 | BL | IndexProg    | Date    | Time     | Pedig  | a       | b       | c       | alpha  | beta    | gamma  | Q(A)    | Q(B)     | Q(C)    | Q(D)  | Q(E)    | Q(F)  |
|-----|-------|----------|------|----|--------------|---------|----------|--------|---------|---------|---------|--------|---------|--------|---------|----------|---------|-------|---------|-------|
| 20  | 95.42 | 1759.294 | 1.00 | P  | FJZNV6.22a   | 16Sep02 | 00:23:38 | 141    | 19.8778 | 8.1933  | 11.2407 | 90.000 | 106.056 | 90.000 | 27.4048 | 148.9638 | 85.6992 | .0000 | 26.8072 | .0000 |
| 20  | 95.42 | 1759.294 | 1.00 | P  | FJZNV6.22a   | 16Sep02 | 00:23:38 | 123    | 19.8778 | 8.1933  | 11.2407 | 90.000 | 106.056 | 90.000 | 27.4048 | 148.9638 | 85.6992 | .0000 | 26.8072 | .0000 |
| 20  | 95.42 | 1759.294 | 1.00 | P  | FJZNV6.22a   | 16Sep02 | 00:23:38 | 122    | 19.8778 | 8.1933  | 11.2407 | 90.000 | 106.056 | 90.000 | 27.4048 | 148.9638 | 85.6992 | .0000 | 26.8072 | .0000 |
| 20  | 95.41 | 1759.294 | 1.00 | P  | FJZNV6.22a   | 16Sep02 | 00:23:38 | 143    | 19.8778 | 8.1933  | 11.2407 | 90.000 | 106.056 | 90.000 | 27.4048 | 148.9638 | 85.6992 | .0000 | 26.8072 | .0000 |
| 20  | 95.41 | 1759.294 | 1.00 | P  | FJZNV6.22a   | 16Sep02 | 00:23:38 | 121    | 19.8778 | 8.1933  | 11.2407 | 90.000 | 106.056 | 90.000 | 27.4048 | 148.9638 | 85.6992 | .0000 | 26.8072 | .0000 |
| 20  | 88.1  | 1760.24  | 1.00 | P  | ITO12/log    | 16Sep02 | 00:22:14 | 353    | 19.8806 | 8.1960  | 11.2414 | 90.000 | 106.057 | 90.000 | 27.397  | 148.866  | 85.688  | .000  | 26.802  | .000  |
| 20  | 86.3  | 1760.27  | 1.00 | P  | ITO12/log    | 16Sep02 | 00:22:14 | 342    | 19.8808 | 8.1960  | 11.2415 | 90.000 | 106.057 | 90.000 | 27.397  | 148.866  | 85.687  | .000  | 26.802  | .000  |
| 20  | 85.8  | 1760.29  | 1.00 | P  | ITO12/log    | 16Sep02 | 00:22:14 | 153    | 19.8809 | 8.1961  | 11.2415 | 90.000 | 106.057 | 90.000 | 27.396  | 148.863  | 85.687  | .000  | 26.802  | .000  |
| 20  | 85.2  | 1760.25  | 1.00 | P  | ITO12/log    | 16Sep02 | 00:22:14 | 132    | 19.8807 | 8.1960  | 11.2415 | 90.000 | 106.057 | 90.000 | 27.397  | 148.866  | 85.687  | .000  | 26.802  | .000  |
| 20  | 82.3  | 1759.427 | 1.00 | P  | DICVOL91/log | 16Sep02 | 00:25:48 | Mon__4 | 19.8762 | 8.1953  | 11.2391 | 90.000 | 106.046 | 90.000 | 27.406  | 148.892  | 85.714  | .000  | 26.794  | .000  |
| 20  | 82.3  | 1759.427 | 1.00 | P  | DICVOL91/log | 16Sep02 | 00:25:48 | Mon__1 | 19.8762 | 8.1953  | 11.2391 | 90.000 | 106.046 | 90.000 | 27.406  | 148.892  | 85.714  | .000  | 26.794  | .000  |
| 20  | 69.9  | 1759.329 | 1.00 | P  | DICVOL91/log | 16Sep02 | 00:25:48 | Mon__6 | 19.9473 | 8.1958  | 11.2380 | 90.000 | 106.745 | 90.000 | 27.407  | 148.873  | 86.349  | .000  | 28.032  | .000  |
| 20  | 69.9  | 1759.329 | 1.00 | P  | DICVOL91/log | 16Sep02 | 00:25:48 | Mon__3 | 19.9473 | 8.1958  | 11.2380 | 90.000 | 106.745 | 90.000 | 27.407  | 148.873  | 86.349  | .000  | 28.032  | .000  |
| 20  | 58.31 | 7038.717 | 4.00 | C  | KOHLv7.01b   | 16Sep02 | 00:24:21 | 2      | 22.4813 | 16.3899 | 19.8782 | 90.000 | 106.056 | 90.000 | 21.4249 | 37.2261  | 27.4036 | .0000 | 13.4035 | .0000 |
| 20  | 50.73 | 7036.909 | 4.00 | C  | KOHLv7.01b   | 16Sep02 | 00:24:21 | 3      | 22.4787 | 16.3877 | 19.9477 | 90.000 | 106.737 | 90.000 | 21.5803 | 37.2360  | 27.4040 | .0000 | 14.0067 | .0000 |
| 20  | 48.50 | 3518.315 | 2.00 | P  | KOHLv7.01b   | 16Sep02 | 00:24:21 | 5      | 19.8778 | 16.3840 | 11.2415 | 90.000 | 106.055 | 90.000 | 27.4044 | 37.2528  | 85.6860 | .0000 | 26.8038 | .0000 |
| 20  | 31.8  | 1760.251 | 1.00 | P  | DICVOL91/log | 16Sep02 | 00:25:48 | Mon__5 | 19.8748 | 8.1974  | 11.2394 | 90.000 | 105.995 | 90.000 | 27.396  | 148.815  | 85.666  | .000  | 26.698  | .000  |
| 20  | 31.8  | 1760.251 | 1.00 | P  | DICVOL91/log | 16Sep02 | 00:25:48 | Mon__2 | 19.8748 | 8.1974  | 11.2394 | 90.000 | 105.995 | 90.000 | 27.396  | 148.815  | 85.666  | .000  | 26.698  | .000  |

..(soln 29)  
20 5.9 3517.772 2.00 P DICVOL91/log 16Sep02 00:25:48 Ort\_\_1 38.2370 11.2487 8.1786 90.000 90.000 90.000 6.840 79.031 149.498 .000 .000 .000  
..(soln 35)  
19 10.12 3524.489 2.00 A LZONv6.23b 16Sep02 00:27:15 70847 15.4918 20.3323 11.9122 99.309 93.939 72.188 45.9880 27.2859 72.3962 13.1721 2.3608 -21.2132

Relevant indexing log entries:

\*\* FJZN6 on RR2: Crysfire 2002 v9.45.25 QDAT-file, 16Sep02 at 00:23:36  
RR2 = Sample 2 - sdpdrr2 - Wavelength 0.79764 (23-11-1998)  
First 40 obs lines (out of 150), wavelength = 0.7976400  
Volmax not required, Z2Theta correction = 0.0 deg  
Special FJZN6 parameters: (none)

FJZN v6.22a run started on 16Sep02 at 00:23:37  
RR2 = Sample 2 - sdpdrr2 - Wavelength 0.79764 (23-11-1998)  
15 solutions found with I20>15 and Merit > 7.0  
I20 Merit Volume V/V1 BL IndexProg Date Time Pedig a b c alpha beta gamma Q(A) Q(B) Q(C) Q(D) Q(E) Q(F)  
20 95.42 1759.294 1.00 P FJZNV6.22a 16Sep02 00:23:38 123 19.8778 8.1933 11.2407 90.000 106.056 90.000 27.4048 148.9638 85.6992 .0000 26.8072 .0000  
20 95.42 1759.294 1.00 P FJZNV6.22a 16Sep02 00:23:38 141 19.8778 8.1933 11.2407 90.000 106.056 90.000 27.4048 148.9638 85.6992 .0000 26.8072 .0000  
20 95.42 1759.294 1.00 P FJZNV6.22a 16Sep02 00:23:38 122 19.8778 8.1933 11.2407 90.000 106.056 90.000 27.4048 148.9638 85.6992 .0000 26.8072 .0000  
20 95.41 1759.294 1.00 P FJZNV6.22a 16Sep02 00:23:38 121 19.8778 8.1933 11.2407 90.000 106.056 90.000 27.4048 148.9638 85.6992 .0000 26.8072 .0000  
20 95.41 1759.294 1.00 P FJZNV6.22a 16Sep02 00:23:38 143 19.8778 8.1933 11.2407 90.000 106.056 90.000 27.4048 148.9638 85.6992 .0000 26.8072 .0000

Chekcell (10Oct02)

Using cell 1 from the above SUM file (FJZN6 soln 1)

I20 Merit Volume V/V1 BL IndexProg Date Time Pedig  
20 95.42 1759.294 1.00 P FJZNV6.22a 16Sep02 00:23:38 141  
  
a b c alpha beta gamma  
19.8778 8.1933 11.2407 90.000 106.056 90.000  
  
Q(A) Q(B) Q(C) Q(D) Q(E) Q(F)  
27.4048 148.9638 85.6992 .0000 26.8072 .0000

150 obs lines

Optimise tolerance gives 0.019 deg 2Theta

Best Group (using Tol=0.019)

Sp. Group Sigma Indexed Calculated  
P2 0.00277 150 365  
P21 0.00277 150 363  
C2 0.00398 100 181  
PM 0.00277 150 365  
PC 0.00328 139 315  
PA 0.00328 139 315  
CM 0.00398 100 181  
CC 0.00302 94 156  
CA 0.00302 94 156  
P2/M 0.00277 150 365  
P21/M 0.00277 150 363  
C2/M 0.00398 100 181  
P2/C 0.00328 139 315  
P21/A 0.00378 141 312  
P21/C 0.00328 139 313  
P21/N 0.00279 150 312  
C2/C 0.00302 94 156  
Best estimated group(s):  
P21/N 0.00279 150 312

Truecell did not improve this, nor LePage as far as it was taken (run aborted after several minutes)

The results were judged to be of sufficient quality not to need further refinement.

This cell and spacegroup was accepted as correct, as described in the summary above

\*\* End of outline of indexing procedure \*\*

# SDPD\_RR3

RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)

Synchrotron data, capillary sample

\*\* Summary of Results \*\*

Crystal system: Tetragonal

Unit cell (from TREOR, M19 = 268, no 2Theta zero correction thought necessary):

|         |         |        |        |        |        |          |
|---------|---------|--------|--------|--------|--------|----------|
| a       | b       | c      | alpha  | beta   | gamma  | Volume   |
| 13.3546 | 13.3546 | 9.4421 | 90.000 | 90.000 | 90.000 | 1683.944 |

Systematic absences condition(s):

hhl: l = 2n

Spacegroup(s) consistent with this condition:

No. 105, P42mc  
No. 112, P-42c  
No. 131, P4(2)/mmc

41 lines indexed out of 42 observed (as reported by PowderX)  
using 2Theta tolerance 0.02 deg

Unindexed line(s):

Line 20 at 2Theta = 16.251 deg (assumed to be from another solid phase)

\*\*\*\*\*

\*\* Outline of indexing procedure \*\*

Crysfire 2002 v9.45.25 (17Sep02)

Self-calibration (Z2Th)

Suggested correction = +0.003 deg (not applied)

| I20 | Merit | Volume   | V/V1 | BL | IndexProg    | Date    | Time     | Pedig  | a       | b       | c       | alpha  | beta   | gamma  | Q(A)     | Q(B)     | Q(C)     | Q(D)  | Q(E)  | Q(F)  |
|-----|-------|----------|------|----|--------------|---------|----------|--------|---------|---------|---------|--------|--------|--------|----------|----------|----------|-------|-------|-------|
| 20  | 25.6  | 3368.668 | 1.00 | P  | DICVOL91/log | 17Sep02 | 02:37:12 | Tet__1 | 13.3543 | 13.3543 | 18.8894 | 90.000 | 90.000 | 90.000 | 56.074   | 56.074   | 28.026   | .000  | .000  | .000  |
| 20  | 17.42 | 3373.748 | 1.00 | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Tet__4 | 18.9046 | 18.9046 | 9.4401  | 90.000 | 90.000 | 90.000 | 27.9810  | 27.9810  | 112.2143 | .0000 | .0000 | .0000 |
| 20  | 17.39 | 3373.785 | 1.00 | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Tet__3 | 18.9047 | 18.9047 | 9.4401  | 90.000 | 90.000 | 90.000 | 27.9808  | 27.9808  | 112.2134 | .0000 | .0000 | .0000 |
| 20  | 15.82 | 3373.585 | 1.00 | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Tet__1 | 13.3666 | 13.3666 | 18.8821 | 90.000 | 90.000 | 90.000 | 55.9703  | 55.9703  | 28.0480  | .0000 | .0000 | .0000 |
| 20  | 15.80 | 3373.620 | 1.00 | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Tet__2 | 13.3667 | 13.3667 | 18.8821 | 90.000 | 90.000 | 90.000 | 55.9699  | 55.9699  | 28.0478  | .0000 | .0000 | .0000 |
| 20  | 15.38 | 1543.874 | .46  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__5 | 6.1285  | 9.4446  | 26.6733 | 90.000 | 90.000 | 90.000 | 266.2519 | 112.1078 | 14.0556  | .0000 | .0000 | .0000 |
| 20  | 14.9  | 3367.817 | 1.00 | P  | DICVOL91/log | 17Sep02 | 02:37:12 | Tet__2 | 18.8967 | 18.8967 | 9.4314  | 90.000 | 90.000 | 90.000 | 28.005   | 28.005   | 112.421  | .000  | .000  | .000  |
| 20  | 13.70 | 2030.153 | .60  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__9 | 8.0584  | 9.4436  | 26.6775 | 90.000 | 90.000 | 90.000 | 153.9947 | 112.1316 | 14.0510  | .0000 | .0000 | .0000 |
| 20  | 13.52 | 2031.151 | .60  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__4 | 8.0614  | 9.4440  | 26.6794 | 90.000 | 90.000 | 90.000 | 153.8791 | 112.1216 | 14.0490  | .0000 | .0000 | .0000 |
| 20  | 13.33 | 2031.318 | .60  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__7 | 8.0621  | 9.4440  | 26.6793 | 90.000 | 90.000 | 90.000 | 153.8522 | 112.1214 | 14.0492  | .0000 | .0000 | .0000 |
| 20  | 12.28 | 1685.160 | .50  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__8 | 6.6889  | 9.4449  | 26.6740 | 90.000 | 90.000 | 90.000 | 223.5069 | 112.0995 | 14.0547  | .0000 | .0000 | .0000 |
| 20  | 11.95 | 1942.668 | .58  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__3 | 7.7020  | 9.4401  | 26.7188 | 90.000 | 90.000 | 90.000 | 168.5729 | 112.2148 | 14.0076  | .0000 | .0000 | .0000 |
| 20  | 11.45 | 1943.354 | .58  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__6 | 7.7039  | 9.4397  | 26.7230 | 90.000 | 90.000 | 90.000 | 168.4934 | 112.2237 | 14.0033  | .0000 | .0000 | .0000 |
| 20  | 9.53  | 1667.390 | .49  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__1 | 9.4446  | 13.2059 | 13.3686 | 90.000 | 90.000 | 90.000 | 112.1068 | 57.3413  | 55.9534  | .0000 | .0000 | .0000 |
| 20  | 6.21  | 2239.334 | .66  | P  | TAUPv3.3a    | 17Sep02 | 02:33:45 | Ort__2 | 5.9179  | 13.3576 | 28.3286 | 90.000 | 90.000 | 90.000 | 285.5412 | 56.0461  | 12.4609  | .0000 | .0000 | .0000 |

19 268 1683.944 .50 P TREOR90/log 17Sep02 02:42:03 Tet\_\_1 13.3546 13.3546 9.4421 90.000 90.000 90.000 56.0712 56.0712 112.1671 .0000 .0000 .0000

From this it's obvious that we have a high-symmetry cell, probably tetragonal.

Relevant indexing log entries:

\*\* TAUP on RR3: Crysfire 2002 v9.45.26 QDAT-file, 17Sep02 at 02:33:39  
RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)  
First 20 obs lines (out of 42), wavelength = 0.7976400  
Volmax = 6000.0000 (default), Z2Theta correction = 0.0 deg  
Special TAUP parameters: Del2Th = 0.040 (default), Nspuri = 0 (default)  
Search down to orthorhombic (default), X off (V < 125% Vlow, default)

TAUP v3.3a run started on 17Sep02 at 02:33:45

RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)

13 solutions found (using steering codes "DFG CTHO")

| I20 | Merit | Volume   | V/V1 | BL | IndexProg | Date    | Time     | Pedig  | a       | b       | c       | alpha  | beta   | gamma  | Q(A)     | Q(B)     | Q(C)     | Q(D)  | Q(E)  | Q(F)  |
|-----|-------|----------|------|----|-----------|---------|----------|--------|---------|---------|---------|--------|--------|--------|----------|----------|----------|-------|-------|-------|
| 20  | 17.42 | 3373.748 | 1.00 | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Tet__4 | 18.9046 | 18.9046 | 9.4401  | 90.000 | 90.000 | 90.000 | 27.9810  | 27.9810  | 112.2143 | .0000 | .0000 | .0000 |
| 20  | 17.39 | 3373.785 | 1.00 | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Tet__3 | 18.9047 | 18.9047 | 9.4401  | 90.000 | 90.000 | 90.000 | 27.9808  | 27.9808  | 112.2134 | .0000 | .0000 | .0000 |
| 20  | 15.82 | 3373.585 | 1.00 | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Tet__1 | 13.3666 | 13.3666 | 18.8821 | 90.000 | 90.000 | 90.000 | 55.9703  | 55.9703  | 28.0480  | .0000 | .0000 | .0000 |
| 20  | 15.80 | 3373.620 | 1.00 | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Tet__2 | 13.3667 | 13.3667 | 18.8821 | 90.000 | 90.000 | 90.000 | 55.9699  | 55.9699  | 28.0478  | .0000 | .0000 | .0000 |
| 20  | 15.38 | 1543.874 | .46  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__5 | 6.1285  | 9.4446  | 26.6733 | 90.000 | 90.000 | 90.000 | 266.2519 | 112.1078 | 14.0556  | .0000 | .0000 | .0000 |
| 20  | 13.70 | 2030.153 | .60  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__9 | 8.0584  | 9.4436  | 26.6775 | 90.000 | 90.000 | 90.000 | 153.9947 | 112.1316 | 14.0510  | .0000 | .0000 | .0000 |
| 20  | 13.52 | 2031.151 | .60  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__4 | 8.0614  | 9.4440  | 26.6794 | 90.000 | 90.000 | 90.000 | 153.8791 | 112.1216 | 14.0490  | .0000 | .0000 | .0000 |
| 20  | 13.33 | 2031.318 | .60  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__7 | 8.0621  | 9.4440  | 26.6793 | 90.000 | 90.000 | 90.000 | 153.8522 | 112.1214 | 14.0492  | .0000 | .0000 | .0000 |
| 20  | 12.28 | 1685.160 | .50  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__8 | 6.6889  | 9.4449  | 26.6740 | 90.000 | 90.000 | 90.000 | 223.5069 | 112.0995 | 14.0547  | .0000 | .0000 | .0000 |
| 20  | 11.95 | 1942.668 | .58  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__3 | 7.7020  | 9.4401  | 26.7188 | 90.000 | 90.000 | 90.000 | 168.5729 | 112.2148 | 14.0076  | .0000 | .0000 | .0000 |
| 20  | 11.45 | 1943.354 | .58  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__6 | 7.7039  | 9.4397  | 26.7230 | 90.000 | 90.000 | 90.000 | 168.4934 | 112.2237 | 14.0033  | .0000 | .0000 | .0000 |
| 20  | 9.53  | 1667.390 | .49  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__1 | 9.4446  | 13.2059 | 13.3686 | 90.000 | 90.000 | 90.000 | 112.1068 | 57.3413  | 55.9534  | .0000 | .0000 | .0000 |
| 20  | 6.21  | 2239.334 | .66  | P  | TAUPv3.3a | 17Sep02 | 02:33:45 | Ort__2 | 5.9179  | 13.3576 | 28.3286 | 90.000 | 90.000 | 90.000 | 285.5412 | 56.0461  | 12.4609  | .0000 | .0000 | .0000 |

TAUP v3.3a run finished on 17Sep02 at 02:33:45 (Elapsed time: .93 sec)

\*\* DICVOL91 on RR3: Crysfire 2002 v9.45.26 QDAT-file, 17Sep02 at 02:37:08

RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)

First 20 obs lines (out of 42), wavelength = 0.7976400

Volmax = 6000.0000 (default), Z2Theta correction = 0.0 deg

Special DICVOL91 parameters: Del2Th = 0.030 (default), Volmin = 0 (default)

Search high systems (cub/tet/hex)

DICVOL91 post-processor DVLOG v2.1 started on 17Sep02 at 02:37:12

RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)

2 solutions reported:

| I20 | Merit | Volume   | V/V1 | BL | IndexProg    | Date    | Time     | Pedig  | a       | b       | c       | alpha  | beta   | gamma  | Q(A)   | Q(B)   | Q(C)    | Q(D) | Q(E) | Q(F) |
|-----|-------|----------|------|----|--------------|---------|----------|--------|---------|---------|---------|--------|--------|--------|--------|--------|---------|------|------|------|
| 20  | 25.6  | 3368.668 | 1.00 | P  | DICVOL91/log | 17Sep02 | 02:37:12 | Tet__1 | 13.3543 | 13.3543 | 18.8894 | 90.000 | 90.000 | 90.000 | 56.074 | 56.074 | 28.026  | .000 | .000 | .000 |
| 20  | 14.9  | 3367.817 | 1.00 | P  | DICVOL91/log | 17Sep02 | 02:37:12 | Tet__2 | 18.8967 | 18.8967 | 9.4314  | 90.000 | 90.000 | 90.000 | 28.005 | 28.005 | 112.421 | .000 | .000 | .000 |

Elapsed time in DICVOL91 down to orthorhombic: .440 sec

(DICVOL91 did not report the elapsed time down to monoclinic)

Total elapsed time reported by DICVOL91: .440 sec

DVLOG run finished on 17Sep02 at 02:37:12

\*\* TREOR90 on RR3: Crysfire 2002 v9.45.26 QDAT-file, 17Sep02 at 02:42:02

RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)

First 25 obs lines (out of 42), wavelength = 0.7976400

Volmax = 6000.0000 (default), Z2Theta correction = 0.0 deg

Special TREOR90 parameters: Del2Th = 0.040 (default)

TREOR90 post-processor TRLOG v1.2 started on 17Sep02 at 02:42:03

RR3 = Sample 3 - sdpdrr2 - Wavelength 0.79764 (0-1-1916)

1 solutions reported (up to 25 obs lines, max cell side = 25.0, max volume = 6000.0)

| I20 | Merit | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  | a       | b       | c      | alpha  | beta   | gamma  | Q(A)    | Q(B)    | Q(C)     | Q(D)  | Q(E)  | Q(F)  |
|-----|-------|----------|------|----|-------------|---------|----------|--------|---------|---------|--------|--------|--------|--------|---------|---------|----------|-------|-------|-------|
| 19  | 268   | 1683.944 | 1.00 | P  | TREOR90/log | 17Sep02 | 02:42:03 | Tet__1 | 13.3546 | 13.3546 | 9.4421 | 90.000 | 90.000 | 90.000 | 56.0712 | 56.0712 | 112.1671 | .0000 | .0000 | .0000 |

(TREOR90 does not report elapsed times)

TRLOG run finished on 17Sep02 at 02:42:03

Chekcell (10Oct02)

Using cell 16 from SUM file (TREOR90 soln 1)

| I20 | Merit | Volume   | V/V1 | BL | IndexProg   | Date    | Time     | Pedig  |
|-----|-------|----------|------|----|-------------|---------|----------|--------|
| 19  | 268   | 1683.944 | .50  | P  | TREOR90/log | 17Sep02 | 02:42:03 | Tet__1 |

| a       | b       | c      | alpha  | beta   | gamma  |
|---------|---------|--------|--------|--------|--------|
| 13.3546 | 13.3546 | 9.4421 | 90.000 | 90.000 | 90.000 |

| Q(A)    | Q(B)    | Q(C)     | Q(D)  | Q(E)  | Q(F)  |
|---------|---------|----------|-------|-------|-------|
| 56.0712 | 56.0712 | 112.1671 | .0000 | .0000 | .0000 |

42 obs lines

Optimise tolerance gives 0.146 deg 2Theta (incl unindexed line 20 at 16.25 deg)  
Not wishing to be constrained by the iunindexed line, used Tol = 0.02 deg

Best Group (using Tol=0.02)

| Sp. Group | Sigma   | Indexed | Calculated |
|-----------|---------|---------|------------|
| P4        | 0.00010 | 41      | 172        |
| P41       | 0.00010 | 41      | 167        |
| P42       | 0.00010 | 41      | 169        |
| P43       | 0.00010 | 41      | 167        |
| I4        | 0.00005 | 24      | 85         |
| I41       | 0.00005 | 24      | 83         |
| P-4       | 0.00010 | 41      | 172        |
| I-4       | 0.00005 | 24      | 85         |
| P4/M      | 0.00010 | 41      | 172        |
| P42/M     | 0.00010 | 41      | 169        |
| P4/N      | 0.00006 | 38      | 155        |
| P42/N     | 0.00006 | 38      | 152        |
| I4/M      | 0.00005 | 24      | 85         |
| I41/A     | 0.00005 | 23      | 75         |
| P422      | 0.00010 | 41      | 172        |
| P4212     | 0.00009 | 40      | 168        |
| P4122     | 0.00010 | 41      | 167        |
| P41212    | 0.00010 | 41      | 167        |
| P4222     | 0.00010 | 41      | 169        |
| P42212    | 0.00009 | 40      | 165        |
| P4322     | 0.00010 | 41      | 167        |
| P43212    | 0.00009 | 40      | 163        |
| I422      | 0.00005 | 24      | 85         |
| I4122     | 0.00005 | 24      | 83         |
| P4MM      | 0.00010 | 41      | 172        |
| P4BM      | 0.00012 | 37      | 150        |
| P42CM     | 0.00013 | 38      | 150        |
| P42NM     | 0.00014 | 38      | 148        |
| P4CC      | 0.00014 | 38      | 137        |
| P4NC      | 0.00015 | 38      | 135        |
| P42MC     | 0.00011 | 41      | 156        |
| P42BC     | 0.00012 | 37      | 134        |
| I4MM      | 0.00005 | 24      | 85         |
| I4CM      | 0.00005 | 22      | 75         |
| I41MD     | 0.00005 | 23      | 76         |
| I41CD     | 0.00005 | 21      | 66         |
| P-42M     | 0.00010 | 41      | 172        |
| P-42C     | 0.00011 | 41      | 156        |
| P-421M    | 0.00009 | 40      | 168        |
| P-421C    | 0.00010 | 40      | 152        |
| I-4M2     | 0.00005 | 24      | 85         |
| P-4C2     | 0.00013 | 38      | 150        |



|                          |         |    |     |
|--------------------------|---------|----|-----|
| P-4B2                    | 0.00012 | 37 | 150 |
| P-4N2                    | 0.00014 | 38 | 148 |
| P-4M2                    | 0.00010 | 41 | 172 |
| I-4C2                    | 0.00005 | 22 | 75  |
| P-42M                    | 0.00010 | 41 | 172 |
| I-42D                    | 0.00005 | 23 | 76  |
| P4/MMM                   | 0.00010 | 41 | 172 |
| P4/MCC                   | 0.00014 | 38 | 137 |
| P4/NBM                   | 0.00007 | 33 | 137 |
| P4/NNC                   | 0.00010 | 34 | 122 |
| P4/MBM                   | 0.00012 | 37 | 150 |
| P4/MNC                   | 0.00015 | 38 | 135 |
| P4/NMM                   | 0.00006 | 38 | 155 |
| P4/NCC                   | 0.00009 | 35 | 120 |
| P42/MMC                  | 0.00011 | 41 | 156 |
| P42/MCM                  | 0.00013 | 38 | 150 |
| P42/NBC                  | 0.00008 | 33 | 121 |
| P42/NNM                  | 0.00010 | 34 | 135 |
| P42/MBC                  | 0.00012 | 37 | 134 |
| P42/MNM                  | 0.00014 | 38 | 148 |
| P42/NMC                  | 0.00007 | 38 | 139 |
| P42/NCM                  | 0.00008 | 35 | 133 |
| I4/MMM                   | 0.00005 | 24 | 85  |
| I4/MCM                   | 0.00005 | 22 | 75  |
| I41/AMD                  | 0.00005 | 22 | 71  |
| I41/ACD                  | 0.00005 | 20 | 61  |
| Best estimated group(s): |         |    |     |
| P42MC                    | 0.00011 | 41 | 156 |
| P-42C                    | 0.00011 | 41 | 156 |
| P42/MMC                  | 0.00011 | 41 | 156 |

\*\* Conclusions (as shown in the summary above) \*\*

Crystal system:

Tetragonal

Unit cell:

| a       | b       | c      | alpha  | beta   | gamma  | Volume   |
|---------|---------|--------|--------|--------|--------|----------|
| 13.3546 | 13.3546 | 9.4421 | 90.000 | 90.000 | 90.000 | 1683.944 |

Systematic absences condition(s):

hhl: l = 2n

Spacegroup(s) consistent with this condition:

No. 105, P42mc  
 No. 112, P-42c  
 No. 131, P4(2)/mmc

41 lines indexed out of 42 observed (as reported by PowderX)  
 using 2Theta tolerance 0.02 deg

Unindexed line(s):

Line 20 at 2Theta = 16.251 deg (assumed to be from another solid phase)

\*\* End of outline of indexing procedure \*\*