Reexamination of KNO₃ IV

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should be more or less hydrostatic at high pressures, say 10 kb or above, but this may not be true at 3 or 5 kbar.

It was recently found that Cu radiation will penetrate beryllium sufficiently to give x-ray diffraction effects of samples under high pressures in the "squeezer" apparatus. Although the pattern of $KNO_3 IV$ (see Fig. 3) obtained with Cu radiation was much weaker than one obtained with molybdenum radiation, it nevertheless sufficed for a refinement of the data obtained in the cylinder. Using several remanent low-pressure lines from a record obtained with copper radiation taken before completion of the transition, several high-pressure spacings were calculated and used to correct the other data. The final tabulation of corrected data is found in Table 1.

It was observed that initially there was considerable extrusion of sample in the "squeezer" due to shearing forces. With the sample under pressure for several hours to two or three days, the pattern improved in sharpness and intensity, and the low-pressure lines weakened considerably or even disappeared entirely. The latter effect is probably due to an increase of the pressure homogeneity with time as the beryllium anvil creeps and becomes dished. The pressure is calculated from the area of the carboloy face (5 mm) since the initial beryllium pellet increases in diameter to this maximum. Using 1° slits and the correct beryllium pellet size, one can leave a sample at a pressure of 9 kbar for days without the sample height changing enough to reduce the pattern intensity noticeably.

Results

A. Indexing

Our first procedure was to compare with the Bell-Austin charts (Battelle Mem. Inst.) for possible hexagonal indexing, but no fit was found. Application of the Hesse-Lipson procedure with elimination of possible axial sets on density considerations yielded the final cell data. Indices and cell constants for the high-pressure modification were calculated and refined with the aid of an IBM 7090 computer. The final axial values determined are $a = 11.04_8$ Å, $b = 8.36_7$ Å, $c = 7.40_2$ Å with a:b:c: = 1.320:1:0.885 (Z = 10).

One line appeared to be a doublet on the molybdenum record and this was substantiated upon examination of the copper record. Only on the latter are the lines indexed as 030 and 400 clearly resolved. In spite of this discovery and also the discovery of a few weak lines not observed by JAMLESON, the pattern can still be indexed with axial

| $\texttt{Table 1.} X \text{-} ray diffraction data on KNO_3 IV; a = \! 11.04_8 \text{\AA}, b = \! 8.36_7 \text{\AA}, c = \! 7.40_2 \text{\AA}, Z = \! 10$ | | | | |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|---------------|-----|-----------------------|
| hkl | d_{obs} | $d_{ m calc}$ | | $100 \frac{I^*}{I_0}$ |
| 020 | 4.18 Å | 4.18 Å | | 6 |
| 002 | 3.70 | 3.70 | | 29 |
| 310 | 3.37 | 3.37 | 2 | 3 |
| 112 | 3.25 | 3.24 | | 5 |
| 202 | 3.08 | 3.08 | | 100 |
| 030 | 2.80 | 2.79 | | 1 |
| 022 | | 2.77 | | |
| 400 | 2.76 | 2.76 | | \$ 95 |
| 320 | | 2.76 | | |
| 122 | 2.68 | 2.69 | | 53 |
| 230 | 2.49 | 2.49 | 1 | . 95 |
| 222 | | 2.48 | 1 | |
| 103 | 2.421 | 2.408 | | 13 |
| 113 | 2.314 | 2.314 | | 37 |
| 420 | | 2.305 | 1 | |
| 402 | 2.213 | 2.213 | 1 | 69 |
| 322 | | 2.214 | | 00 |
| 412 | 2.141 | 2.140 | 1 | 83 |
| 510 | | 2.136 | | |
| 232 | 2.071 | 2.066 | | 7 |
| 140 | | 2.056 | | |
| 033 | 1.848 | 1.848 | | 12 |
| 512 | | 1.850 | | |
| 323 | × 1 | 1.841 | | |
| 600 | | 1.841 | | |
| 333 | 1.652 | 1.652 | | 9 |
| 304 | | 1.653 | | |
| 150 | | 1.654 | | |
| 532 | 1.572 | 1.569 | | 12 |
| 700 | | 1.578 | - 1 | |
| 251 | | 1.565 | | |
| 334 | 1.421 | 1.422 | | 20 |
| 632 | | 1.419 | 1 | 10 - 0 |
| 504 | | 1.419 | | |
| 533 | | 1.418 | | |
| 524 | 1.343 | 1.343 | | 7 |
| 244 | | 1.344 | - 1 | , |
| 811 | | 1.340 | | |
| 434 | | 1.346 | | |
| 253 | | 1.338 | | |
| 812 | 1.281 | 1.279 | | 4 |
| 361 | | 1.284 | | T |
| 732 | | 1.288 | | |
| | | | 1 | |

* Integrated intensities.

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