Reexamination of KNO₃ IV

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four-ton ram mounted on the base of the 6-inch press upon which is set either the "squeezer" or the supported beryllium cylinder.

Both $CuK\alpha$ and $MoK\alpha$ radiation were used in the study. The former was used only with the "squeezer" inasmuch as the thick walls of the cylinder reduced the beam strength considerably.

The sample was prepared by mixing 50 per cent KNO_3 and 50 per cent starch (by volume) and compressing the mixture into a 1-mm pellet by means of a pellet press. The pellet was pushed to the bottom of the cylinder bore and covered with a second pellet of compressed



Fig. 2. A. KNO₃ II at room pressure, MoK α radiation. The large peak between ten and eleven degrees is actually a doublet, 111 and 021, not resolved. Supported beryllium cylinder; B. KNO₃ IV at 3.7 kb, MoK α radiation. Note the two peaks only slightly resolved at about 14.3 degrees. These are 030 and 400 of the high-pressure form. Supported beryllium cylinder. Attenuation 4-1-8. Pellet 4.6 \times 1.0 mm diam.

beryllium powder, the latter serving to separate the sample reflecting surface from the steel or carboloy piston. Disks cut from beryllium rod may also be used, but because of creep, cause excessive friction at higher pressures. Not all $\rm KNO_3$ pellets were half starch, but such a mixture tends to give more complete conversion in a shorter time than does the pure material alone. The pattern obtained with the pure material, however, shows somewhat sharper lines.

Figure 2 shows the low-pressure modification (KNO₃ II), and $KNO_3 IV \text{ at } 3.7 \text{ kbar}$ (room temperature). These patterns were obtained in the supported vessel with piston and sample size of 4.6 mm, using

MoK α radiation and Zr filter. Scanning speed is $\frac{1}{4}$ °/min; scale factor of 4; 8-sec time constant; and chart speed of $\frac{1}{8}$ in./min. Earlier results with the diamond anvil showed some of these high-pressure peaks. Unfortunately three moderately strong beryllium lines are also present in the pattern and may even conceal some of the pattern. None of the lines observed by JAMIESON are covered, however. It should be pointed out that this particular pattern was obtained after the sample had remained under this same pressure overnight. No low-pressure peaks are seen to remain.



Fig. 3. A. $\text{KNO}_3 \Pi$ at room pressure. $\text{Cu}K\alpha$ radiation. Note the partially resolved 111 and 021 peaks at 23+ degrees, that were completely unresolved in Fig.2A. Bridgman anvil device with beryllium pellets; B. $\text{KNO}_3 \text{IV}$ at 6.6 kbar (nominal), $\text{Cu}K\alpha$ radiation. Note the completely resolved 030 and 400 peaks at 32 degrees that were only partially resolved in Fig.2B. Bridgman anvil device with beryllium pellet. Attenuation 4-1-8. Pellet 5.0 mm diam.

with beryman penet. Attendation 4-1-6. 1 ener 5.0 min diam.

The data used for the indexing were obtained from a run using undiluted KNO₃ (Baker's Analyzer, ACS standard) and the sample was scanned at $\frac{1}{8}$ °/min, chart speed of $\frac{1}{8}$ in./min. Initially the lines were corrected for sample-height change by the position of a low-pressure line on the chart before conversion was complete. A compressibility of 5×10^{-6} /bar was used to correct the shift of the low-pressure line due to compression up to the transition pressure. The assumption is made that when both low- and high-pressure phases are present the pressure on the remaining low-pressure phase is less than or equal to the equilibrium pressure. In the supported pressure vessel the pressure

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