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The samples submitted from Berkeley have been examined under the microscope and, except possibly for some needles from benzene just arrived here, only the inactive form from aqueous alcohol contained single crystals large enough for oscillation photographs.

One of these crystals (the inactive form from aqueous alcohol) was mounted and examined on the goniometer and appeared to be monoclinic. Some oscillation photographs confirmed this finding but also showed that Cu K $\alpha$  radiation is not suitable for examining these compounds. It is strongly absorbed by the cobalt and reradiated as fluorescent Co K radiation, which badly fogs the films.

Radiation from any target metal of lower atomic number than copper will be satisfactory and it is desirable to stay as close to copper as possible in order that the rays will not be too soft. Either nickel or cobalt appeared suitable and since a cobalt target was available on one of our tubes work was continued with Co K radiation filtered through 0.001" iron foil to remove Co K $\beta$ . Since suitable vacuum cameras were not available for this tube, a target blank was ordered from the shop for the vacuum outfit. This has now been plated, after trying several techniques, with a layer of cobalt metal which has so far stood up satisfactorily through about two hundred hours of operation. Test powder photographs of metallic copper have been made to check the purity of the spectrum obtained and only lines due to Co K $\alpha$  could be seen even after long exposures. Only a few pictures of the active compound in vacuum have recently been obtained with this outfit.

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BY AUTH. CG DAR-1 431

DATE 4/1/96

W. E. Frank DRADA 3/1/96

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The inactive compound is monoclinic with  $b_0 = 7.00 \text{ \AA}$ .  $a_0$  or  $a/2$  is  $7.30 \text{ \AA}$  and  $c_0$  or  $c/2$  is  $14.15 \text{ \AA}$ . The exact length of these latter, if desired, will require oscillation photographs about these axes. The angle  $\beta$  is about  $115^\circ$ . The crystals are mostly thin plates with roughly hexagonal outline, sometimes elongated along  $b_0$  into flat stubby needles. The larger individuals are poorly formed, bent and badly stuck together or twinned.

The pyridinate has crystallites which, although too small to handle satisfactorily, are still quite large and require pulverization and rotation to yield good powder picture lines. They are much greater than  $10^{-3} \text{ cm}$ .

The active compound, both with and without oxygen, and prepared either by Rumford or University of California, has much smaller particle size. The samples need not be ground or rotated to yield good powder lines. The particles must be less than  $10^{-3} \text{ cm}$  in size. A lower limit cannot be set with pictures made so far. The oxygenated compounds from the two sources yield pictures which appear identical both as to spacing and intensities of the lines, except for two very definite lines which appear on the pictures of the Rumford product and not on the other. It is very probable that the Rumford product contains several percent of some impurity. These two lines correspond in spacing to the two strongest lines of the graphite pattern but one cannot positively identify an impurity on the basis of only two lines. It is planned to prepare pictures of the U. of C. product containing small quantities of added graphite for comparison. If other possible impurities are suggested we shall test them also.

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The oxygenated and oxygen-free compounds yield pictures very similar in the large spacing (small scattering angle) region but quite different in the smaller spacing (larger scattering angle) regions. Pictures of partially oxygenated compound will be required to determine whether there is a fundamental change in structure on adding oxygen or merely a small change of unit cell dimensions. Fairly small changes in unit cell edge-lengths and angles might account for a complete change in the appearance of the pattern at higher scattering angles. If the crystals from benzene prove to be single crystals of the active compound it will greatly facilitate the settling of this question.

Since most of the pictures so far prepared have been of a trial nature in a non-vacuum camera, copies of the photographs and detailed measurements are not being submitted.

July 6, 1942 .

Signed

Edward W. Hughes  
Edward W. Hughes

Approved

Linus Pauling  
Linus Pauling

cc:

2 to Calvin  
1 to Pauling  
1 to Ed Hughes

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