THE CRYSTAL STRUCTURE OF COBALtic ACETYLACETONATE Co (C₅H₇O₂)₃

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(Morgan (1926) has reported that the metallic acetylacetonates exhibit some peculiar properties. For instance, the metallic atom no longer shows ionic properties which are used for its detection in chemical analysis. To account for this, he assumed that (C₅H₇O₂) radical is a chelate group. With the metal atom it has plane configuration for the ring system. The stability of the metallic acetylacetonate is ascribed to the symmetrical arrangement of three chelate groups round the metallic atom which corresponds to the arrangement of octahedral co-ordination of six oxygen atoms around the central atom. Astbury (1926) has made a preliminary study of these tervalent metal acetylacetonates. So far no detailed structure work of cobaltic acetylacetonate has been reported in literature. The present paper reports the complete structure analysis by X-ray diffraction.

EXPERIMENTAL

The crystals were prepared by the method described by Barbierri (1928). The unit cell dimensions as determined from rotation and Weissenberg photographs are a = 14.16, b = 7.48, c = 16.43 Å and β = 98° 41'. These values are in good agreement with those reported by Astbury. Taking the density to be 1.43, the number of molecules per unit cell is 4. There were no systematic absences in the hkl reflections; the list of reflecting planes reveals that (0k0) is absent when k is odd and (l0l) when l is odd. The space-group is therefore C₆h₈ — P 2₁/c. Using the multiple film technique, the zero layer Weissenberg photographs were taken on suitable crystal specimens rotating about the three axes using Cu-Kα radiation. The intensities of the reflections were estimated visually and corrected in the usual way for the Lorentz and polarisation factors. Using Wilson's method, the intensities were put on an absolute scale, which were later further corrected by comparison with the calculated values. This Wilson's method also gave a factor B = 2.4 Å² for temperature correction. The crystals used were about
-015 mm. thickness and the correction for absorption was made following Bradley (1940).

**DETERMINATION OF THE STRUCTURE**

As there are four cobalt atoms in the unit cell, it was considered desirable to study Patterson projections to locate these heavy atoms. Using relative values of $F^2$, Patterson projections on $(ac)$ and $(bc)$ planes were made. These projections gave the following parameters for the cobalt atom of a single molecule,

\[ x = 0.26; \quad y = 0.16; \quad z = 0.24. \]

The projections did not clearly indicate the Co-O position. With these parameters a trial electron density projection on $(ac)$ plane was made by using the signs obtained from the 'heavy atom'. The first projection clearly indicated the positions of three oxygen atoms and ten carbon atoms. Following the suggestion of Morgan that the acetylacetone radical is a planar ring configuration, a series of refinements were made. Figure 1 shows the electron density projection of $\text{Co(C}_2\text{H}_5\text{O)}_3$ on $(ac)$ plane, showing one molecule. Contour scale arbitrary.