previously been described (Delf, 1963). This method is based on the approach due to Pike & Wilson (1959) but without the use of horizontal truncation. The only significant difference between this and the method of Taylor *et al.* (method D of Parrish *et al.*) is that, while both are symmetric-limit methods, the method described by Taylor *et al.* uses a symmetric wavelength range while that of Delf uses a symmetric angular range.

There may be a theoretical advantage in using symmetric wavelength ranges, but it is less convenient than symmetric angular ranges in practice, as line profiles are invariably recorded as angular intensity distributions in X-ray powder diffraction measurements. In practice the two methods will only differ significantly at high angles, where the centroid approach is unlikely to be of use because of the large dispersion effect and the non-linearity, and increase in magnitude, of the background level.

There has been a certain amount of criticism of the centroid method of determining line positions as some truncation procedure has to be used to overcome the difficulties introduced by the Cauchy-like tails. It is, therefore, of interest that two laboratories have independently converged on essentially the same truncation procedure to give a reproducible measure of line position which can easily be related to the wavelength scale.

References

Delf, B. W. (1963). *Brit. J. Appl. Phys.* **14**, 345.

Parrish, W., Mack, M. & Taylor, J. (1963). *J. Appl. Phys.* **34**, 2544.

Pike, E. R. & Wilson, A. J. C. (1959). *Brit. J. Appl. Phys.* **10**, 57.

Taylor, J., Mack, M. & Parrish, W. (1963). *Acta Cryst.* **16**, 1179.

Acta Cryst. (1964). **17**, 771

Hydrogen bonding in (+)-demethanolaconinone hydroiodide trihydrate.

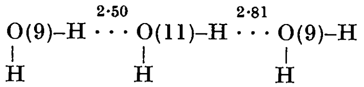
By Jerry Donohue,
Department of Chemistry, University of Southern California, Los Angeles 7, California, U.S.A.

(*Received* 21 *November* 1963)

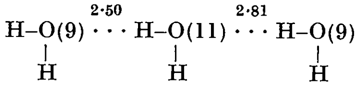
The crystal structure of (+)-demethanolaconinone hydroiodide trihydrate, $C_{24}H_{31}O_4(OH)_4N.HI.3H_2O$, was determined by Przybylska (1961). A water molecule-iodine chain in the structure was somewhat later described by Clark (1963), who listed the hydrogen bonds and angles associated with that chain, and who quoted from a private communication from Przybylska as follows: *Considering the very low ionic character of* HI *and the fact that the iodine atom is situated at a distance of* 5 Å *from the nitrogen atom of the alkaloid, it seems unlikely that the hydrogen of* HI *is attached to nitrogen.* This suggestion cannot be accepted, however, because it ignores the fact that the alkaloid is a moderately strong organic base, and HI a strong acid, indeed, the strongest of the hydrogen halides.

Rather than discuss in detail the essentially incomplete suggestions of Przybylska (1961) and Clark (1963) regarding the hydrogen bonding, we shall instead discuss it from first principles in order to locate, if possible, the hydrogen atoms. This can be done in the following stepwise manner, with the help of Table 2 of Clark (1963):

1. We note first that there are eleven hydrogen atoms available for hydrogen bond formation: six water, four hydroxyl, and one $\geqslant N^+–H$.
2. There are five $I^- \cdots O$ distances in the range 3·52–3·62 Å. These are assumed to correspond to $O–H \cdots I^-$. Three of the oxygen atoms are hydroxyl. This fixes the hydrogen atoms of O(5), O(6), and O(7), and one each on the water oxygen atoms O(9) and O(10).
3. There are five $O \cdots O$ distances in the range 2·50–2·84 Å. These are assumed to correspond to $O–H \cdots O$.
4. One of these involves O(6), whose hydrogen atom is already assigned elsewhere. This gives $O(10)–H \cdots O(6)$.
5. Both hydrogens of O(10) are now assigned. This gives $O(11)–H \cdots O(10)$.
6. The hydroxyl O(8) is involved in only one short $O \cdots O$ system. This gives $O(8)–H \cdots O(9)$.
7. Eight of the ten hydrogen atoms on oxygen have now been placed. For the remaining two there is an ambiguity:

either 

$$\underset{\underset{\mathrm{H}}{|}}{\mathrm{O(9)}}\!-\!\mathrm{H}\overset{2{\cdot}50}{\cdots}\underset{\underset{\mathrm{H}}{|}}{\mathrm{O(11)}}\!-\!\mathrm{H}\overset{2{\cdot}81}{\cdots}\underset{\underset{\mathrm{H}}{|}}{\mathrm{O(9)}}\!-\!\mathrm{H}$$

or 

$$\mathrm{H}\!-\!\underset{\underset{\mathrm{H}}{|}}{\mathrm{O(9)}}\overset{2{\cdot}50}{\cdots}\mathrm{H}\!-\!\underset{\underset{\mathrm{H}}{|}}{\mathrm{O(11)}}\overset{2{\cdot}81}{\cdots}\mathrm{H}\!-\!\underset{\underset{\mathrm{H}}{|}}{\mathrm{O(9)}}$$

8. The above considerations are made solely with regard to interatomic distances. *This criterion is not enough,* since it ignores covalent bond angles involving hydrogen. However, reference to Table 3 of Clark (1963) shows that all of the $C–O \cdots I$, $C–O \cdots O$, $O \cdots O \cdots O$ and $O \cdots O \cdots I$ angles implied by the above considerations are within the range of expectation for $C–OH \cdots I$, $C–OH \cdots O$, $O \cdots HOH \cdots O$, and $O \cdots HOH \cdots I$ interactions.
9. Finally, search for a hydrogen bond acceptor for the $\geqslant N^+–H$ group (and there is no reason why this should be an iodine ion) reveals an O(5) of a neighboring molecule at 3·05 Å, and with acceptable $C{\cdot}N \cdots O$ angles.

All of the foregoing, including the ambiguity of item 7 above, are shown in Fig. 1. It is concluded that, on the basis of interatomic distances and presumed interbond angles, nine of the eleven hydrogen atoms of the hydrogen bonds can be positioned. In the case of the remaining two, there is not a marked preference between the two possible arrangements, as may be seen from the values of the relevant angles in Fig. 1.

The discussion of Clark (1963) was mainly concerned with the hydrogen bonding involving the water molecules.

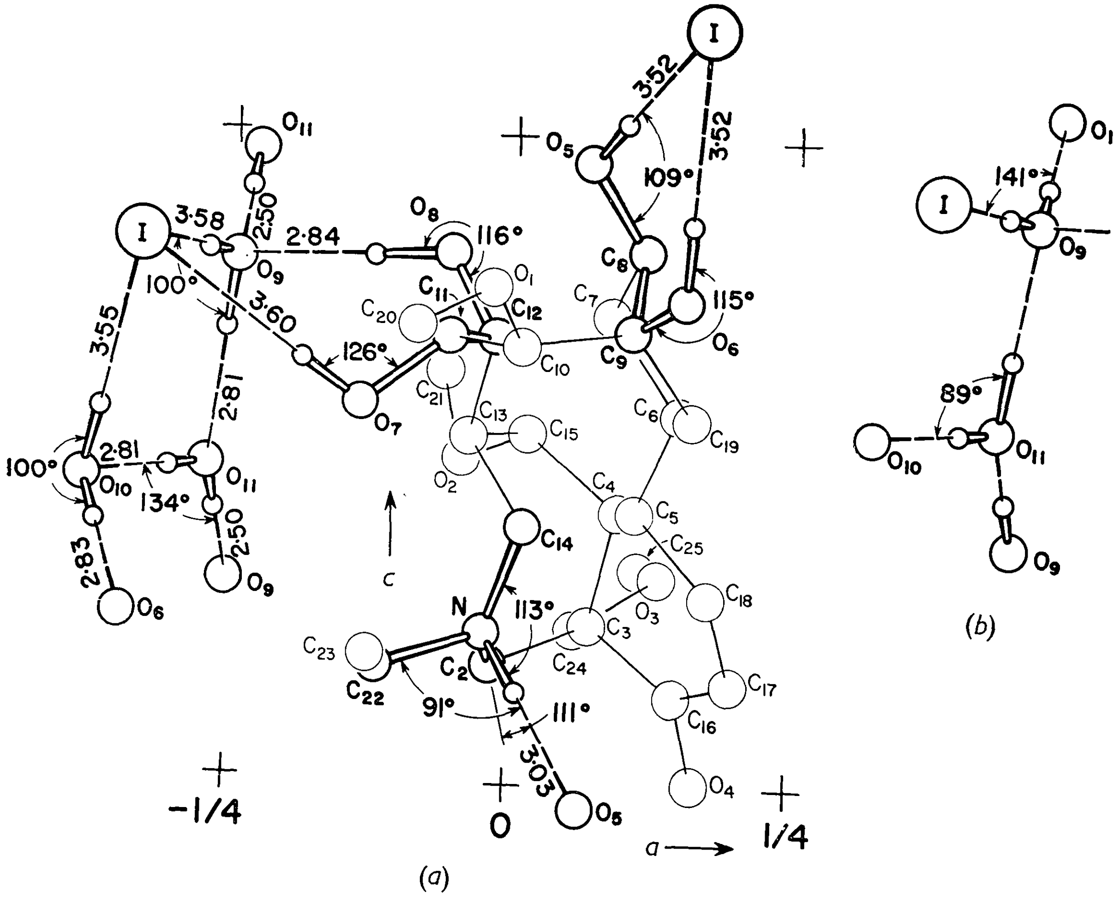


Fig. 1. (a) Hydrogen bonding scheme, showing the locations of the hydrogen atoms. (The true positions of these atoms may not correspond to precisely linear hydrogen bonds, as depicted.) (b) Alternate, and only slightly less favorable, arrangement of the hydrogen atoms of the water molecules O(9) and O(11).

The present note completes that discussion by including the rest of the hydrogen bond donor and acceptor atoms. It is comforting that in this complicated structure the principles governing hydrogen bond formation (Donohue, 1952) are nicely adhered to. Finally, the 'very low' ionic character in an isolated HI molecule is not germane to this alkaloid salt.

This work was supported by a grant from the U.S. Army Research Office.

References

Clark, J. R. (1963). *Acta Cryst.* **16**, 702.
Donohue, J. (1952). *J. Phys. Chem.* **56**, 502.
Przybylska, M. (1961). *Acta Cryst.* **14**, 429.

Acta Cryst. (1964). **17**, 772

Powder indexing and lattice constants for ThC_2.

By P. K. Gantzel and N. L. Baldwin, *General Atomic Division of General Dynamics Corporation, John Jay Hopkins Laboratory for Pure and Applied Science, San Diego, California, U.S.A.*

(*Received* 20 *January* 1964)

In the course of studying phase relationships between some metal carbides, it was desirable to have a set of indices for the powder pattern of ThC_2. A single-crystal study carried out by Hunt & Rundle (1951) provided the necessary information to calculate the powder pattern for ThC_2. However, the calculated *d* spacings varied sufficiently from our observed pattern to necessitate successive trial indices. An X-ray pattern was recorded with the use of Cr *Kα* radiation in a 114·6 mm Straumanis-loading Debye-Scherrer camera filled with hydrogen.