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Optical-diffraction methods for deducing the effects of hydrogen atoms on X-ray intensities.

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In a re-investigation of the structure of triphenylene (Taylor, 1954)* based upon the data of Klug (1950) there appeared to be some indications of the hydrogen atoms on the (001) Fourier projection. These indications were not particularly clear, and there was some doubt whether the work involved in calculating the contributions of the twelve hydrogen atoms to all the $hk0$ reflexions would be worth while. A compromise was reached by using optical methods (Hanson, Lipson & Taylor, 1953) to decide which reflexions were most greatly affected, and the calculations were confined to these.

The method used was as follows. First, the probable positions of the hydrogen atoms in the unit cell were deduced from the known positions of the carbon atoms and an assumed value of 1.07 Å for the C-H distance (Cochran, 1953). A mask was made in which only these hydrogen atoms were represented, and the diffraction pattern was recorded; a representation of the most prominent features is shown in Fig. 1. By laying the

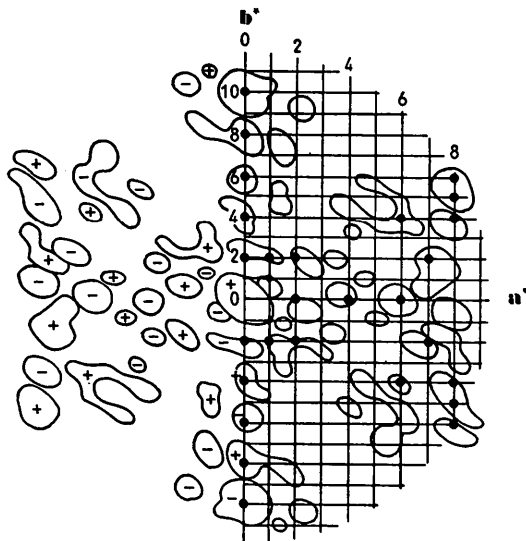


Fig. 1. Representation of prominent features of optical transform of hydrogen atoms in triphenylene. Signs of the transform peaks are shown in the left-hand side and the reciprocal lattice on the right; the reciprocal-lattice points for which the calculations have been carried out are shown by black spots.

reciprocal-lattice net on this diffraction pattern, one could see to which reflexions the hydrogen atoms made the greatest contributions. Table 1 shows the results of calculations for these reflexions in order of increasing θ and it will be seen that the introduction of the hydrogen

atoms does lead to a considerable improvement in the agreement between calculated and observed structure amplitudes.

Optical methods can also be used for providing a rough check on the calculations; by punching an extra hole at the origin, the signs of the hydrogen contributions can

Table 1.

hkl	F_c without hydrogen	F_c with hydrogen	F_o
020	49	45	36
120	68	61	44
200	-82	-73	46
220	-55	-50	40
040	-48	-42	35
400	12	8	9
060	-12	-15	14
600	18	12	11
080	-19	-15	9
640	5	8	8
720	-18	-14	18
0,10,0	18	11	14
840	-23	-25	28
850	18	19	18
860	-11	-10	9

be determined (Lipson & Taylor, 1951), but it is necessary to cover the other 48 holes with gauze in order to enhance the relative effect of this extra hole. One can then tell, without calculation, whether the agreement is improved by introducing the hydrogen atoms.

Some explanation of the symmetry of Fig. 1 is perhaps necessary. Because the plane group is pgg it might be expected that the diffraction pattern should show two planes of symmetry, as the weighted reciprocal lattice does; the contents of one unit cell, however, do not have this symmetry, but the relationship $|F(hk0)| = |F(\bar{h}k0)|$ should obtain at the reciprocal-lattice points. It will be seen that this relationship is reasonably well obeyed over most of the pattern shown in Fig. 1, but there are occasional small discrepancies (e.g. 510 and $\bar{5}10$; 710 and $\bar{7}10$).

The details in Table 1 are provisional: the structure is being refined and work on the (100) projection is proceeding. Also, it may be necessary to introduce an extinction correction, as shown by the systematic discrepancy between F_o and F_c for the first five reflexions.

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References

- COCHRAN, W. (1953). *Acta Cryst.* 6, 260.
 HANSON, A. W., LIPSON, H. & TAYLOR, C. A. (1953). *Proc. Roy. Soc. A*, 218, 371.
 KLUG, A. (1950). *Acta Cryst.* 3, 165.
 LIPSON, H. & TAYLOR, C. A. (1951). *Acta Cryst.* 4, 458.
 TAYLOR, C. A. (1954). In preparation.

* *Editorial note.*—In an article received on 11 November 1953 P. R. Pinnock, C. A. Taylor & H. Lipson pointed out that there was reason to doubt the accuracy of Klug's structure. The paper was subsequently withdrawn pending further experimental investigation.