AN X-RAY STUDY OF FLUORANTHENE CRYSTAL

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INTRODUCTION

Fluoranthene, a crystalline principle isolated from high boiling fraction of coal-tar by the normal process of distillation and repeated fractional crystallization, has the molecular formula $C_{16}H_{10}$ as determined from a study of chemical reactions and formation of various derivatives. Formerly, its molecular formula was given as $C_{15}H_{10}$ by Fittig and Gebhard, but later on Braun and Anton (1929) established uniquely the molecular formula of Fluoranthene as $C_{16}H_{10}$. The present paper reports some new morphological data, differing from those given by Groth and an X-ray investigation of single crystal of Fluoranthene.

EXPERIMENTAL RESULTS AND CONCLUSIONS

Single crystals of Fluoranthene of suitable size were obtained by the slow evaporation of the concentrated solution of the substance in ethyl alcohol. On a morphological examination with the help of a two-circle goniometer, the crystal was found to be monoclinic with (100), (001), ($10\bar{2}$) and (111) faces developing moderately well, but b(010) face being absent. The crystal growth has a tendency to be elongated along b axis. Rotation photographs taken about various possible crystallographic axes were used in determining the fundamental axes. The following Table I gives the results of the complete goniometric study of single crystal of Fluoranthene.

TABLE I

Inter-facial angle of Fluoranthene crystal according to the present author.

$(111) : (\bar{1}11) = 68°$
$(111) : (001) = 86°$
$(\bar{1}02) : (001) = 36° 40'$
$(\bar{1}02) : (\bar{1}01) = 33° 26'$
$(\bar{1}01) : (\bar{1}00) = 51° 39'$
$(100) : (001) = 58° 15'$
$\beta = 121° 45'$

Thus it can be seen that the indices of the so-called m and r faces referred by Groth should be modified to (111) and ($\bar{1}02$). The axis which has been referred by Groth as c axis is really a diagonal axis, i.e. [111], and this is also corroborated by the X-ray rotation photograph taken about this diagonal axis and, consequently, the new monoclinic angle should be 121° 45′. From rotation photographs axial lengths and axial angle were obtained as follows:

$$a = 18{\cdot}46 \text{ Å}$$
$$b = 6{\cdot}205 \text{ Å} \qquad \beta = 121° 45'$$
$$c = 22{\cdot}11 \text{ Å}$$

whence $a : b : c = 2{\cdot}975 : 1 : 3{\cdot}563$.

TABLE II

Indexing of spots and their estimated intensities.
b axis zero-layer

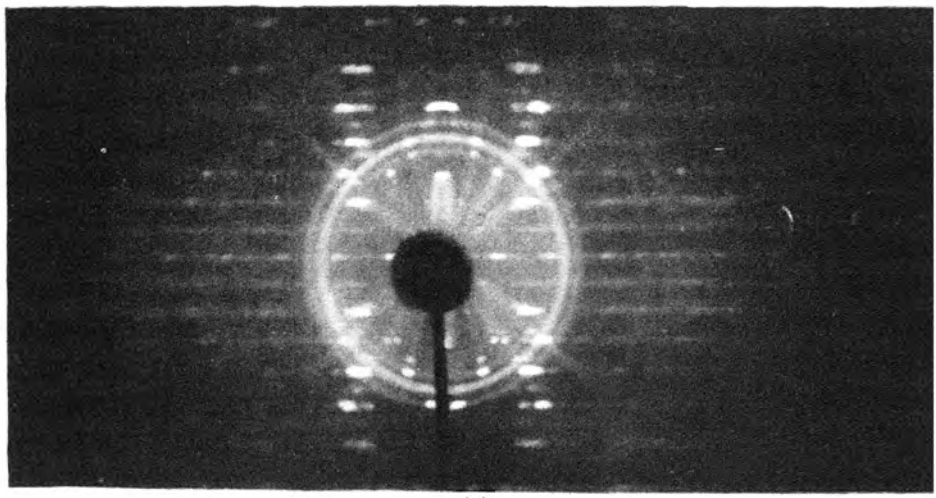
Index of spot	Intensity	Index of spot	Intensity	Index of spot	Intensity
200	m.	20(12)	v.w.	506	m.w.
300	v.s.	20(14)	w.	508	v.w.
500	m.w.	20(16)	w.	50(10)	v.w.
600	m.	302	s.	602	m.
700	m.	304	m.	604	m.
800	m.	306	w.	608	v.w.
900	m.w.	308	m.	702	m.
(10)00	v.w.	30(10)	w.	704	v.w.
(11)00	w.	30(12)	v.w.	802	m.w.
(12)00	v.w.			804	m.
(13)00	v.w.	30(14)	v.w.	902	m.w.
(15)00	v.w.	30(16)	v.w.	906	v.w.
102	m.	402	v.w.	908	w.
104	m.w.	404	m.	90(10)	v.w.
106	m.w.	406	m.	(10)04	v.w.
202	s.	408	m.	(11)02	w.
204	m.w.	40(10)	v.w.	(11)04	v.w.
				(13)02	w.
206	m.	40(12)	v.w.		
208	m.	502	v.w.	(13)04	w.
20(10)	w.	504	m.		

TABLE III

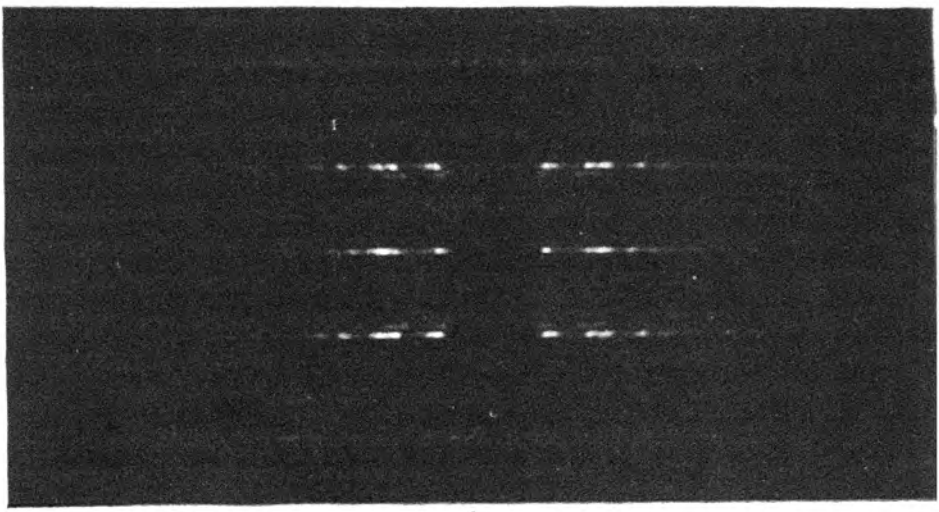
Indexing of spots and their estimated intensities.
a axis zero-layer

Index of spot		Intensity	Index of spot	Intensity	Index of spot	Intensity
002	*	v.s.	020	s.	035	v.w.
004	*	v.s.	040	v.w.	039	v.w.
006	*	s.	06·0	w.	03(10)	v.w.
008	*	s.	021	s.	03(11)	v.w.
00(10)	*	m.	022	m.	041	m.
00(12)	*	w.	023	w.	042	m.
00(14)	*	v.w.	024	s.	043	w.
00(16)	*	w.	026	s.	044	m.w.
012		w.	027	w.	046	v.w.
013		m.	028	m.	047	v.w.
014		w.	029	w.	048	m.w.
015		v.w.	02(10)	m.w.	049	w.
016		v.w.	02(11)	w.	04(11)	m.w.
017		v.w.	02(12)	m.w.	04(12)	w.
018		v.w.	02(13)	m.w.	04(13)	v.w.
019		m.	02(14)	m.w.	04(14)	v.w.
01(11)		v.w.	02(15)	m.w.	062	v.w.
01(12)		v.w.	02(17)	m.w.	063	v.w.
01(13)		v.w.	031	v.w.	065	v.w.
01(15)		w.	032	v.w.	066	v.w.
01(16)		w.	033	v.w.		
			034	v.w.		

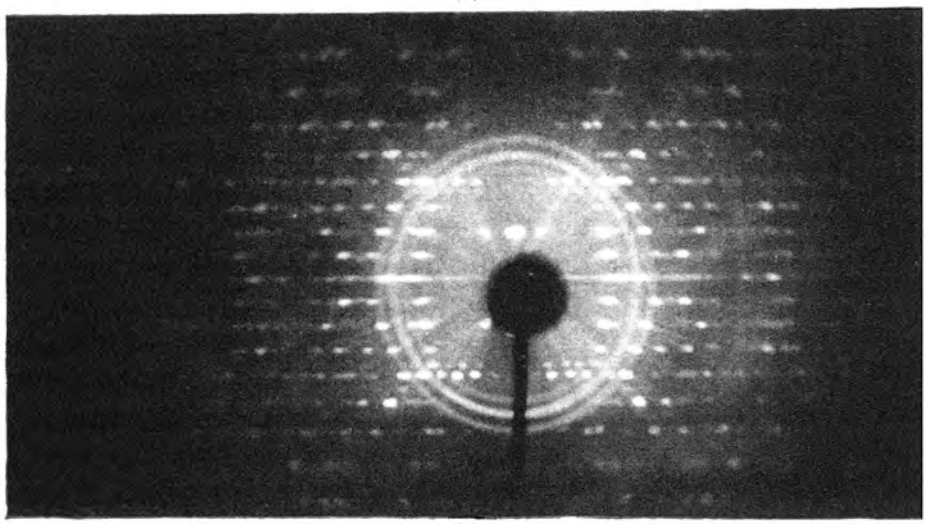
* (00l) planes observed in *a* and *b* axes zero-layer Weissenberg photographs.



(*a*)

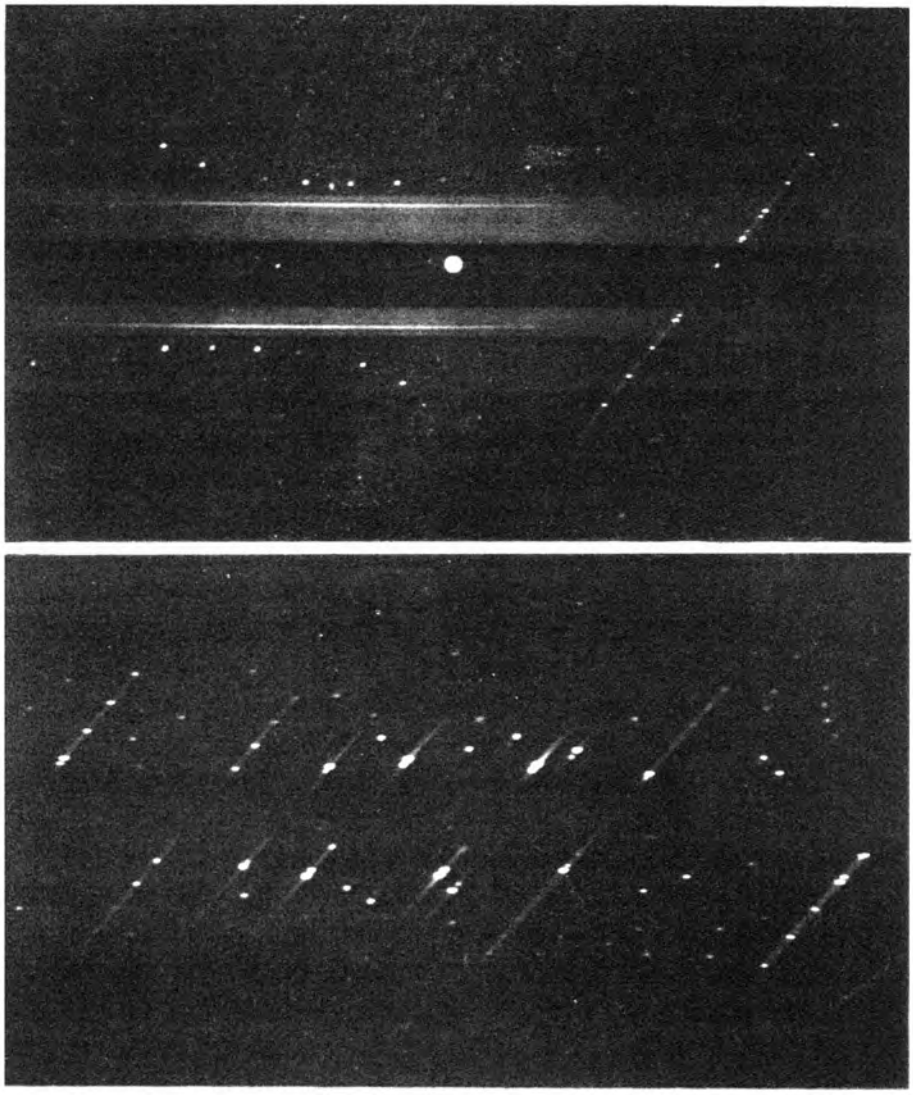


(*b*)



(*c*)

(*a*) Rotating Crystal Photograph taken about *a* axis.
(*b*) Rotating Crystal Photograph taken about *b* axis.
(*c*) Rotating Crystal Photograph taken about *c* axis.
Camera : Cylindrical; radius 4·501 cm.
Radiation : $CuK\alpha$ (unfiltered).



Zero-level Weissenberg Oscillation Photograph taken about a axis (top fig.) and b axis (bottom fig.).

The density of the crystal determined by floatation method was found to be $1{\cdot}238 \pm 0{\cdot}01$ gm. cm.$^{-3}$. On the basis of this value and those already obtained from values of the axial parameters the number of molecules in the unit cell was calculated and found to be 8.

Over-exposed zero-level Weissenberg photographs were taken about b and a axes by the normal-beam method, giving $(h0l)$ and $(0kl)$ reflections respectively. The spots were identified by drawing requisite Weissenberg charts by the method of Schneider (1928). The relative intensities, as determined by the eye-estimation, were noted down against all the spots in Tables II and III.

First and second-level Weissenberg photographs about b axis were also taken by the equi-inclination method, giving $(h1l)$ and $(h2l)$ reflections respectively and the indexing of the spots showed that there were no systematic absence in (hkl) planes.

From Tables II and III it is observed that the conditions of extinction of the spots were

$(h0l)$ planes absent when l is odd.

$(0k0)$ planes absent when k is odd.

The extinction of $h0l$ when l is odd implies the presence of a glide plane parallel to $(0k0)$ with glide component $c/2$. Then the space-group of the crystal definitely contains a glide plane. Again the extinction of $(0k0)$ when k is odd implies that b axis is a two-fold screw axis. So the space-group of the crystal may be written as $P2_1/c$ or C^5_{2h}. Now the number of equivalent point in this space-group is 4, i.e. if one molecule occupies any general position in the unit cell of the crystal, by symmetry operations there should be altogether 4 molecules in the unit cell. But experimentally it has been found that the number of formula weights $C_{16}H_{10}$ in the unit cell is 8. Thus there are two sets of 4 molecules in the unit cell of the crystal, the orientation of the two sets being independent of each other.

Abstract

Single crystal of Fluoranthene with the molecular formula $C_{16}H_{10}$ has been studied both morphologically and by means of X-rays. Goniometric study, together with rotation photographs and density measurement, showed that the crystal was monoclinic with the following axial lengths and axial angle:

$a = 18{\cdot}46$ Å, $b = 6{\cdot}205$ Å, $c = 22{\cdot}11$ Å

$\beta = 121° 45'$

(number of molecules per unit cell = 8)

Weissenberg photographs about the crystallographic axes showed that $(h0l)$ planes are present when l is even and $(0k0)$ plane is present when k is even. The crystal is found to belong to the space-group $C^5_{2h}-P2_1/c$, and so there are four asymmetric pairs of Fluoranthene molecules in the unit cell.

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