

Lab 5135

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X-ray powder patterns and microscopic examination revealed no crystalline impurities.

1. X-Ray Diffraction

Debye-Scherrer diffraction photographs were obtained using filtered copper radiation and a standard Philips 114.6 mm powder camera. The sample was contained in a 0.3 mm Lindemann glass capillary. Observed film readings were corrected for film shrinkage, but not absorption effects. Relative intensities, normalized to give the most intense line a value of 100, were obtained using a Philips X-ray diffractometer and are based on peak heights relative to the base line. The powder pattern is given in Table I.

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TABLE I

TATB POWDER PATTERN

<u>d (meas film)</u>		<u>I/I₁₀₀</u>	<u>Diffractionmeter</u>	<u>hkl</u>	<u>I(precession)</u>	<u>d^a calc from unit cell</u>
7.64	u	16.1	—	110	mw	7.697
7.25		2.1	—	100	vs	7.602
6.03		3.8	—	010	ms	7.213
5.53		2.1	—	011	s	6.026
5.33		5.6	—	101	s	5.511
4.49	u	3.3	—	111	s	5.347
4.362		2.6	—	210	m	4.511
4.269	u	8.3	—	111	s	4.491
4.221	u	8.6	—	101	s	4.365
4.002		3.1	—	111	s	4.276
3.85		---	—	120	s	4.268
3.710		3.5	—	121	s	4.229
--		1.0	—	110	s	4.219
3.600		4.4	—	011	s	4.026
3.385		1.5	—	220	w	3.848
3.138		100.	—	211	s	3.725
			—	201	mw	3.650
			—	211	s	3.603
			—	012	ms	3.397
			—	102	s	3.172
			—	002	vvs	3.137
			—	112	s	3.100
			—	121	m	3.091
			—	122	s	3.047
3.042		2.8	—	111	m	3.037
2.942	u	2.5	—	320	ms	2.952
			—	131	m	2.945
			—	211	ms	2.930
2.833		.90	—	230	s	2.836
2.791		1.1	—	210	m	2.798
2.725		1.7	—	120	ms	2.737
2.609	u	1.0	—	212	m	2.619
			—	122	ms	2.607
2.556	u	1.5	—	301	ms	2.567
			—	330	m	2.566
			—	311	m	2.551
2.534		1.4	—	300	ms	2.534
2.479		1.1	—	232	ms	2.480
2.409	u	0.92	—	032	ms	2.411
			—	030	ms	2.404
2.300		0.41	—	211	m	2.296
2.242		0.50	—	302	ms	2.244
2.205	u	0.55	—	122	m	2.207
			—	322	m	2.199
2.172		0.80	—	112	mw	2.174

TABLE I (Cont.)

<u>d (meas film)</u>	<u>I/I₁₀₀</u>	<u>Diffractometer</u>	<u>hkl</u>	<u>I(precession)</u>	<u>d^d calc from unit cell</u>
2.138 u	6.5	—	421	s	2.147
			222	s	2.138
			240	ms	2.134
			242	s	2.115
2.104 u	6.3	—	220	s	2.110
			322	ms	2.104
			421	s	2.099
2.052 u	0.3	—	133	w	2.060
			203	w	2.045
1.995	0.3		?		
1.928 u	0.42	—	113	w	1.934
			213	mw	1.923
---	0.38	—	412	w	1.907
			103	w	1.906
1.877	0.25		213	w	1.872
1.829	0.25		303	m	1.837
			013	m	1.828
1.787	0.63		251	m	1.788
			252	w	1.759
1.760 u	0.45	—	521	w	1.757
			322	m	1.753
1.68 u	0.25		diffuse (10 lines m-w in intensity)		
1.613	0.19		104	ms	1.613
1.569	2.7		004	vs	1.569
1.548 u	0.67	—	224	ms	1.550
			242	ms	1.545
			244	ms	1.523
1.521 u	0.5	—	222	ms	1.518
			423	ms	1.507
1.502	0.48	—	630		1.504
1.408	0.13		330		1.406
1.302	1.1		661	m	1.303
1.284	0.13		?		
1.215	0.39		?		
1.203	0.25		?		
1.189	0.41		761	m	1.190
1.170	0.14		?		
1.164	0.12		?		
1.114	0.25		?		
1.110	0.29		?		
1.099	0.16		?		
1.087	0.16		?		
1.072	0.09		?		
1.054	0.09		?		
1.032	0.14		?		
1.027	0.14		?		
1.017	0.12		?		
1.008	0.12		?		
0.983	0.20		?		

u = unresolved
 vs = very strong
 s = strong
 ms = medium strong
 m = medium
 mw = medium weak
 w = weak

Single crystal X-ray diffraction photographs were obtained using a precession camera. Unit cell dimensions and angles were calculated from these photographs. The results of these calculations are summarized below.

Space group: P_1 or P_1

Cell dimensions: $a_0 = 9.03 \text{ \AA}$ $\alpha = 108.6^\circ$
 $b_0 = 9.03 \text{ \AA}$ $\beta = 91.8^\circ$
 $c_0 = 6.81 \text{ \AA}$ $\gamma = 120.0^\circ$

Formula weights per cell: 2

Formula weight: 443.12

Density 1.935 (calc); 1.93 ± 0.01 (meas)

2. Optical Crystallography and Other Physical-Chemical Information

The optical crystallography and related information is presented following the outline in Wahlstrom, Optical Crystallography, John Wiley and Sons, Inc., New York (1950), for the examination of nonopaque substances. The relationship between the optical axes and the crystallographic axes are indicated in the figure of a typical TATB crystal. Note that the X optical axis is perpendicular to the a_0b_0 plane so that Y and Z lie in the a_0b_0 plane.

a. Preliminary Megascopic Examination

Color - Light yellow. TATB is both photosensitive and thermally sensitive in that it turns yellowish green on exposure to light, and dark yellow-brown on exposure to elevated temperature.

Specific gravity - 1.93

Fusibility - Not fusible. TATB begins to sublime at about 300°C and to decompose at a somewhat higher temperature. Decomposition occurs without melting and a solid residue remains. A combination of DTA and pyrolysis indicates an exothermic decomposition starting above 300°C. No endothermic transformations are observed.

b. Microscopic Examination

Lower polar only

(1) Morphology

System - Triclinic, pinakoidal

Habit - Tabular, equant, and occasionally imperfect acicular

Forms - $\{001\}$, $\{100\}$, $\{01\bar{1}\}$ on all crystals;
 $\{1\bar{1}\bar{1}\}$ on many crystals;
 $\{011\}$, $\{1\bar{1}\bar{1}\}$ on very few crystals.

(2) Color - Yellow exhibiting strong pleochroism

X = colorless

Y = strong yellow

Z = strong yellow

(3) Luster - Regular reflection off {001} and occasionally off other forms.

(4) Fracture - Uneven or splintery.

(5) Cleavage - Nearly perfect parallel to {001}, and not observed in any other direction.

(6) Refractive indices

Immersion - $X = 1.45$

$$Y > 2.11$$
$$Z > 2.11$$

From estimated 2V and X -

$Y = 2.3$
 $Z = 3.1$

- (7) Inclusions and alternations - No inclusions, but striations parallel to {001} are common.

Crossed Polars

- (1) Anisotropic and biaxial negative
- (2) No twinning
- (3) Abnormal polarization colors
- (4) Dispersion - Rhombic $R > B$
- (5) Optic orientation - see Figure 1
- (6) $2E = 62.8^\circ$
 $2V = 26^\circ$ estimated from centered optic axis figure

Observations on Heating

- (1) Sublimes 300°C and above
- (2) Decomposes without melting $325\text{-}350^\circ\text{C}$ leaving a solid residue

Sublimate

- (1) Lower polar only

- (a) Morphology

- System - Triclinic, pinakoid and pedion faces
 - Habit - Tabular and pyramidal
 - Forms - {001}, others not identified

- (b) Color - Yellow with strong pleochroism

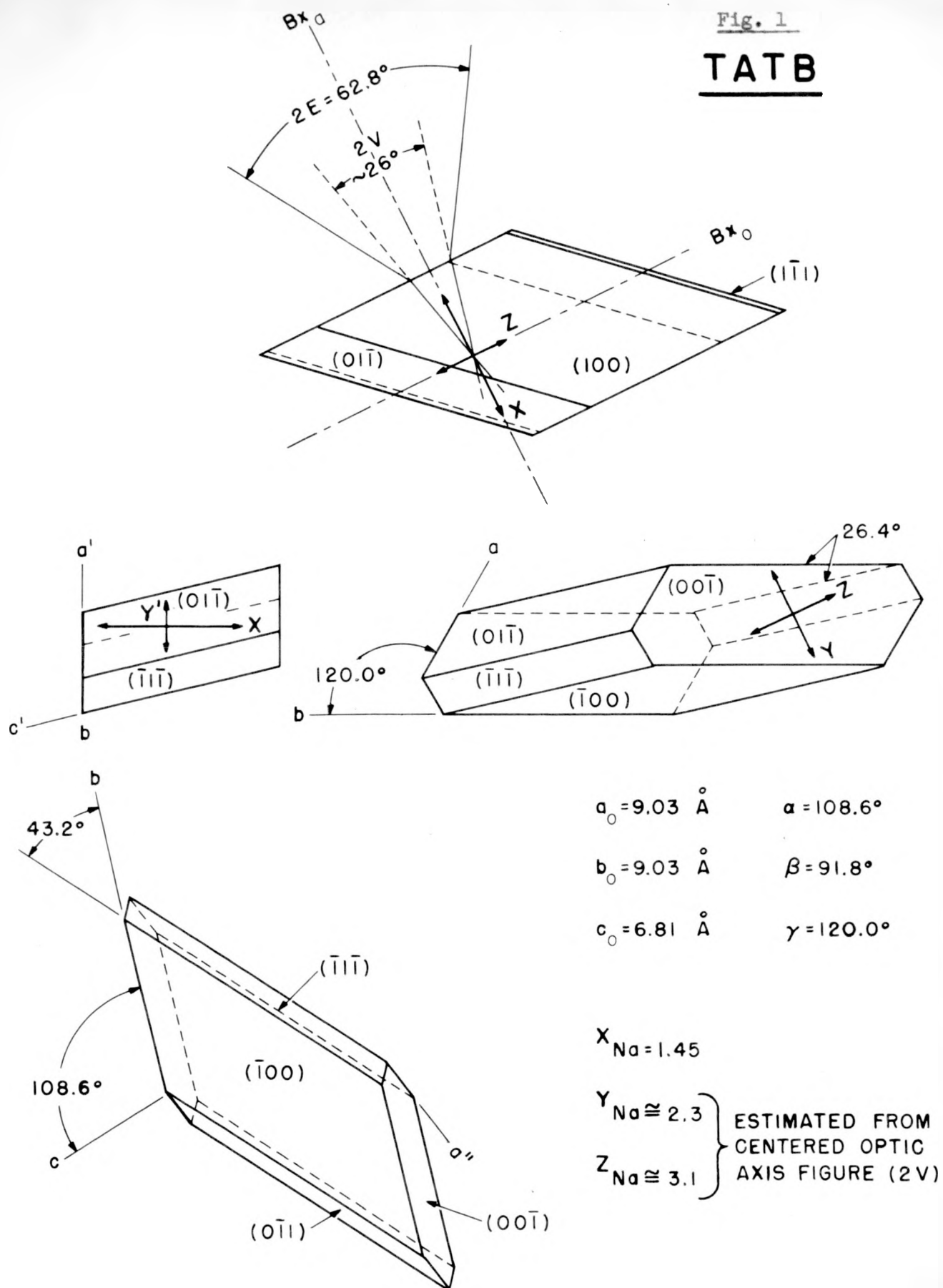
- (c) Index of refraction - same as crystals from nitrobenzene

- (2) Crossed Polars

- (a) Nearly all crystals highly strained and/or twinned.

Fig. 1

TATB



Very poor interference figures with $2E$ varying from 0 to 62.8° .

Optical axial orientation varies from one end of crystal to other.

(b) Anisotropic, biaxial negative in all cases.

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