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CRYSTALLOGRAPHIC DATA

1,3,5-Triamino-2,4,6-trinitrobenzene

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Some of the physical properties and the optical and X-ray crystallography of TATB (1,3,5-triamino-2,4,6-trinitrobenzene) have been investigated at Los Alamos. The following is a summary of the results pertinent to the crystallography.

Crude TATB was prepared by ammonolysis of trichlorotrinitrobenzene. Large crystals of purer TATB, used for the crystallographic studies, were prepared by slow recrystallization from hot nitrobenzene.

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/I<sub>100</sub></u>	<u>Diffractometer</u>	<u>hkl</u>	<u>I (precession)</u>	<u>d<sub>calc</sub> 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
3.042		2.8		002	vvs	3.650
2.942	u	2.5		102	s	3.603
2.833		.90		112	s	3.397
2.791		1.1		121	m	3.172
2.725		1.7		122	s	3.137
2.609	u	1.0		111	m	3.100
2.556	u	1.5		320	ms	3.091
2.534		1.4		131	m	3.047
2.479		1.1		211	ms	3.037
2.409	u	0.92		210	ms	2.952
2.300		0.41		320	m	2.945
2.242		0.50		131	m	2.930
2.205	u	0.55		211	ms	2.836
2.172		0.80		230	s	2.798
				210	m	2.737
				120	ms	2.619
				213	m	2.607
				122	ms	2.567
				301	ms	2.566
				330	m	2.551
				311	m	2.534
				300	ms	2.480
				232	ms	2.411
				032	ms	2.404
				030	ms	2.296
				211	m	2.244
				302	ms	2.207
				122	m	2.199
				322	m	2.174
				112	mw	

TABLE I (Cont.)

<u>d (meas film)</u>	<u>I/I<sub>100</sub></u>	<u>Diffractometer</u>	<u>hkl</u>	<u>I (precession)</u>	<u>d<sub>calc</sub> from unit cell</u>
2.138 u	6.5		421 222 240	s s ms	2.147 2.138 2.134
2.104 u	6.3		242 220 322 421	s s ms s	2.115 2.110 2.104 2.099
2.052 u	0.3		133 203	w w	2.060 2.045
1.995	0.3		?		
1.928 u	0.42		113 213	w mw	1.934 1.923
---	0.38		412 103	w w	1.907 1.906
1.877	0.25		213	w	1.872
1.829	0.25		303 013	m m	1.837 1.828
1.787	0.63		251	m	1.788
1.760 u	0.45		252 521 322	w w m	1.759 1.757 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		224 242	ms ms	1.550 1.545
1.521 u	0.5		244 222	ms ms	1.523 1.518
1.502	0.48		423 630	ms	1.507 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  $P\bar{1}$

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);  $1.93 \pm 0.01$  (meas)

## 2. Optical Crystallography and Other Physical-Chemical Information

The optical crystallography and related information is presented following the outline in Wahllstrom, 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  $a_0 b_0$  plane so that  $Y$  and  $Z$  lie in the  $a_0 b_0$  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}, {011} on all crystals;  
{111} on many crystals;  
{011}, {111} 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  $300^\circ\text{C}$  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

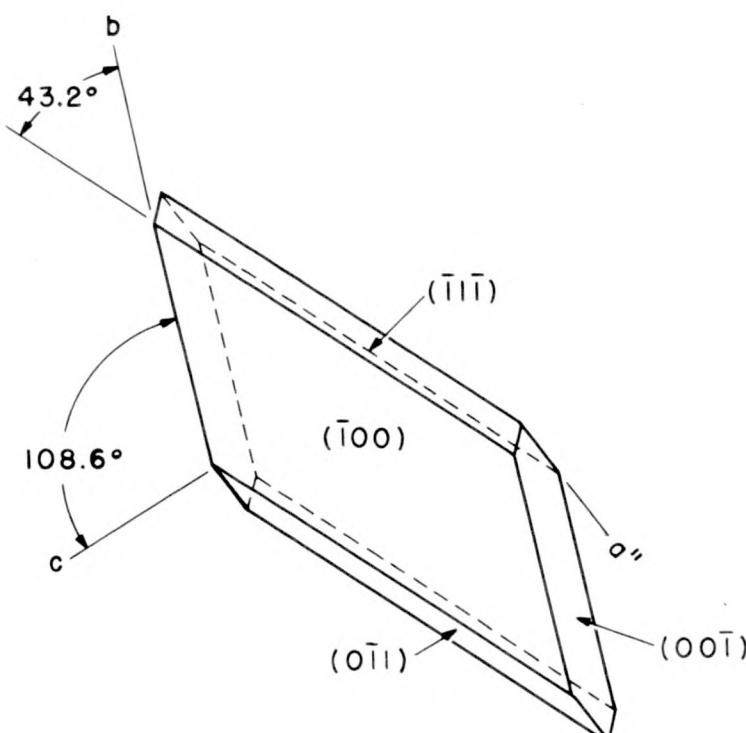
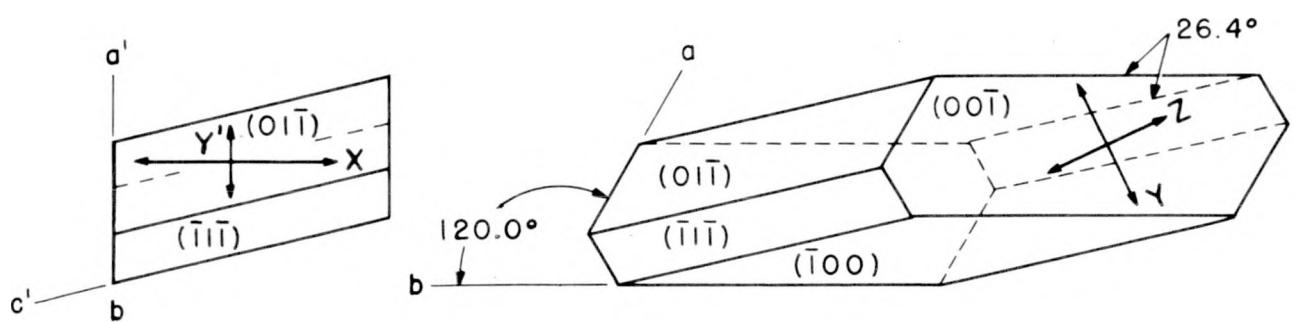
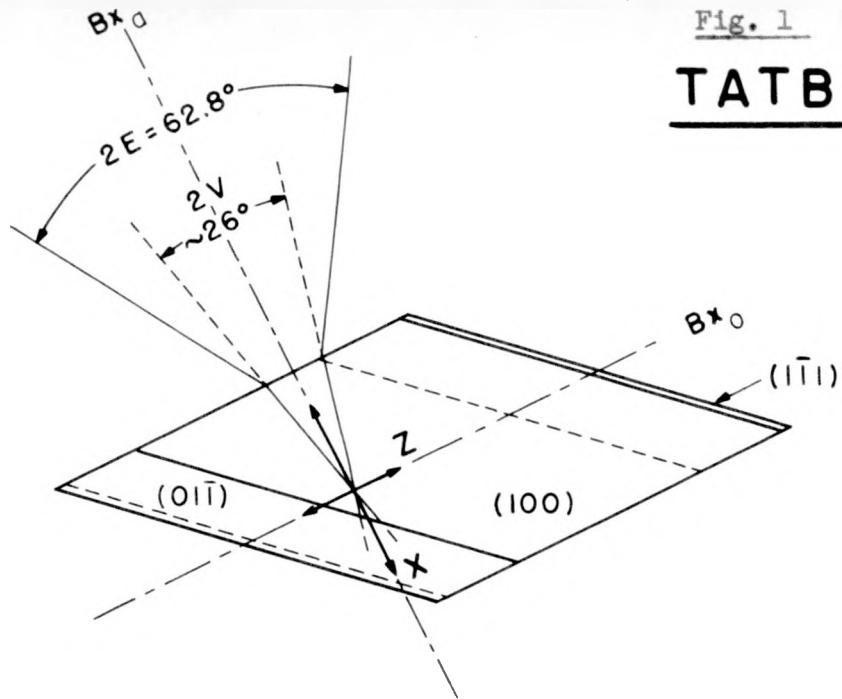
(b) Color - Yellow with strong pleochroism  
(c) Index of refraction - same as crystals from nitrobenzene

(2) Crossed Polars

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

Fig. 1

TATB



$$\begin{array}{ll} a_0 = 9.03 \text{ \AA} & \alpha = 108.6^\circ \\ b_0 = 9.03 \text{ \AA} & \beta = 91.8^\circ \\ c_0 = 6.81 \text{ \AA} & \gamma = 120.0^\circ \end{array}$$

$X_{\text{Na}} = 1.45$   
 $Y_{\text{Na}} \approx 2.3$   
 $Z_{\text{Na}} \approx 3.1$

ESTIMATED FROM  
CENTERED OPTIC  
AXIS FIGURE (2V)

Very poor interference figures with  $2E$  varying from 0 to  $62.8^\circ$ .

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

(b) Anisotropic, biaxial negative in all cases.

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