

terminated as described, any incorporation of activity into the total keto acids less than 0.2% of that of acetate is not significant and probably represents contamination. After paper chromatography more contaminating acetate is removed, and activity in the keto acids greater than 0.05% of the acetate is significant.

ACKNOWLEDGMENT

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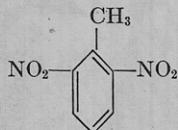
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CRYSTALLOGRAPHIC DATA

89. 2,6-Dinitrotoluene (2,6-DNT)

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Structural Formula for 2,6-Dinitrotoluene, Form I

EXCELLENT crystals of 2,6-dinitrotoluene(I) can be obtained by slow cooling of a hot ethyl alcohol solution with continuous agitation. Benzyl alcohol and thymol are good solvents for recrystallization on a microscope slide (Figure 1). Crystals prepared in this way are orthorhombic and show the forms: prism {110}, basal pinacoid {001}, brachy pinacoid {010}, and brachydome {011}.

2,6-Dinitrotoluene has at least three crystal forms (polymorphs). Form III is apparently unstable at all temperatures from room temperature to the melting point. 2,6-Dinitrotoluene-II is stable above about 40° C. and 2,6-dinitrotoluene(I) is stable below about 40° C.

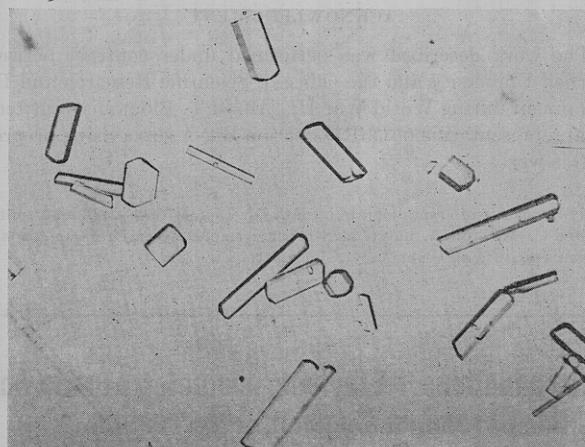


Figure 1. Crystals from Thymol on a Microscope Slide

Well-formed crystals of either I or II can be recrystallized from thymol on a microscope slide. The normal product from fusion is II, although a carefully supercooled melt will nucleate to give III. This very unstable form transforms almost instantaneously, however, to form II, which, in turn, can be seeded with I and slowly transformed to the stable form I.

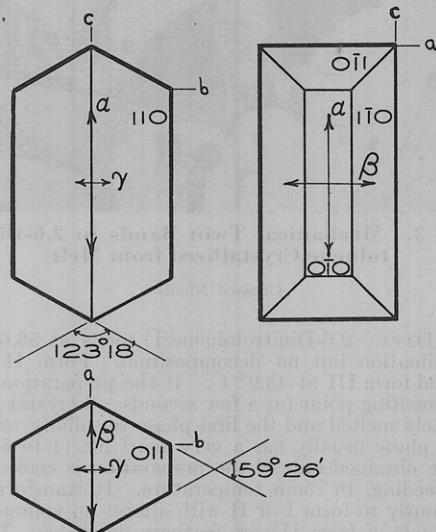


Figure 2. Orthographic Projection of Typical Crystal of 2,6-Dinitrotoluene

CRYSTAL MORPHOLOGY

Crystal System. Orthorhombic.

Form and Habit. Crystals from ethyl alcohol are tablets and rods and show the prism {110}, the brachy pinacoid {010}, and the brachydome {011}. Many crystals from thymol on a microscope slide show the basal pinacoid {001} in addition to the above forms.

Axial Ratio. $a:b:c = 0.571:1:0.539$.

Interfacial Angles (Polar). $110 \wedge \bar{1}\bar{1}0 = 59^\circ 26'$; $011 \wedge \bar{0}\bar{1}1 = 56^\circ 42'$.

X-RAY DIFFRACTION DATA

Cell Dimensions. $a = 7.82$ Å.; $b = 13.70$ Å.; $c = 7.39$ Å.
Formula Weights per Cell. 4 (4.021 calculated from x-ray data).

Formula Weight. 182.13.

Density. 1.536 (flotation in aqueous zinc chloride); 1.528 (x-ray).

Principal Lines

d	I/I_1	d	I/I_1	d	I/I_1
6.86	10	2.70	4	1.786	4
5.18	4	2.64	5	1.760	1
4.98	6	2.58	5	1.735	3
4.21	3	2.51	3	1.688	2
3.90	5	2.44	6	1.625	2
3.67	6	2.32	4	1.589	3
3.56	9	2.29	3	1.567	<1
3.48	5	2.21	<1	1.538	2
3.35	8	2.17	4	1.509	3
3.24	5	2.11	2	1.472	1
3.14	4	2.08	1	1.412	<1
3.10	3	1.996	2	1.330	<1
3.00	2	1.940	1	1.301	<1
2.89	2	1.901	4	1.241	1
2.76	4	1.829	3	1.192	2

OPTICAL PROPERTIES

Refractive Indices (5893 Å.; 25°C.). $\alpha = 1.479 \pm 0.002$. $\beta = 1.697 \pm 0.003$. $\gamma = 1.750 \pm 0.005$.

Optic Axial Angles (5893 Å.; 25°C.). $2V = 53^\circ$ (measured).

$2E = 99^\circ$.

Dispersion. $v > r$.

Optic Axial Plane. 100.

Sign of Double Refraction. Negative.

Acute Bisectrix. $\gamma = b$.

Molecular Refraction (R) (5893 Å.; 25°C.). $\sqrt[3]{\alpha\beta\gamma} = 1.638$.

R (calcd.) = 43.3; R (obsd.) = 42.6.

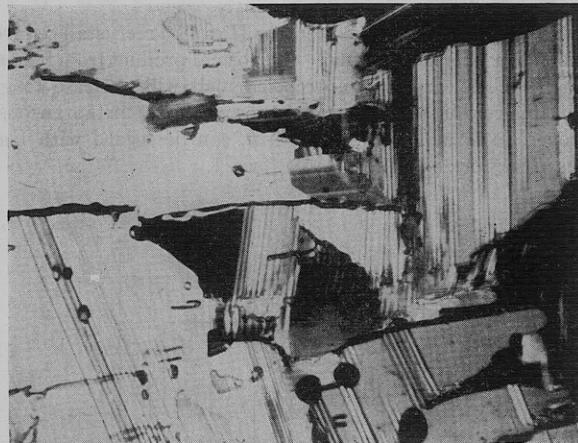


Figure 3. Mechanical Twin Bands in 2,6-Dinitrotoluene Crystallized from Melt

Crossed Nicols

FUSION DATA. 2,6-Dinitrotoluene(I) melts at 56.6°C. with slight sublimation but no decomposition. Form II melts at 64.7°C. and form III at 45.9°C. If the preparation is heated above the melting point for a few seconds, all crystal nuclei will be completely melted and the first phase to solidify will be form III. This phase usually has a very short life (1 to 5 seconds), and can be obtained only if the preparation is cooled, without shock or seeding, to room temperature. It transforms almost instantaneously to form I or II with almost no volume change.

The crystals of form III are feathery dendrites. They show high birefringence with the slow component perpendicular to the direction of most rapid growth. Conoscopic observations show an optic axis near the edge of the field (0.66 N.A.). The sign of double refraction is negative, and the optic axial angle, $2V$, is about 53°.

Form II crystallizes as flat rods showing well-formed, angular ends. Slip bands, produced by mechanical twinning, are shown by the crystals when observed between crossed Nicols. These slip bands are similar to those shown by *p*-dichlorobenzene, but cannot be produced mechanically as easily as they can in *p*-dichlorobenzene. Most of the crystals show oblique extinction of about 30° C. The greater the extinction angle, the nearer one brush approaches the edge of the field. Very rarely a figure showing one optic axis in the field can be obtained. The properties ob-

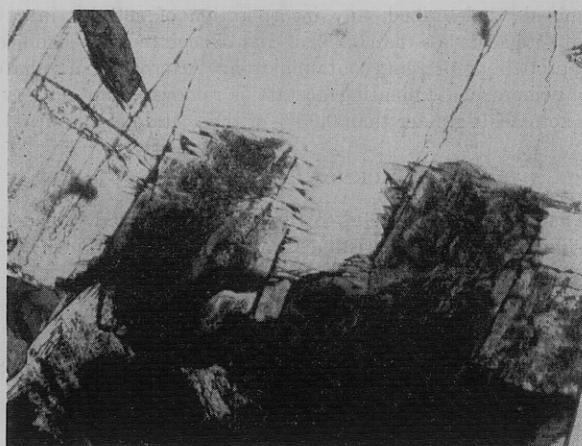


Figure 4. Fusion Preparation of 2,6-Dinitrotoluene Showing II (White) and I Pseudomorphs of II (Dark)

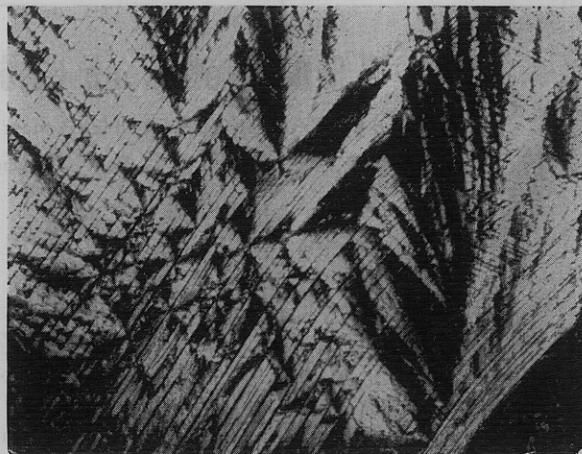


Figure 5. Fusion Preparation of 2,6-Dinitrotoluene Showing II Pseudomorphs of III

tained on this figure are: $2E$ about 90°; (+); $r > v$. Occasional rods that show parallel extinction give a flash figure. On reheating, form I may transform to the higher melting modification, II. It is usually possible, however, to obtain a meltback on crystals of form I. Form II can generally be obtained by scratching form I above the transformation temperature (about 40°C.). When partially remelted, the beta form gives a preparation of pure beta on solidification.

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CONTRIBUTIONS of crystallographic data for this section should be sent to Walter C. McCrone, Analytical Section, Armour Research Foundation of Illinois Institute of Technology, Chicago 16, Ill.

Determination Of Ethylenediaminetetraacetic Acid As the Chromium Complex—Correction

On page 1806 [ANAL. CHEM., 26, 1806 (1954)] the name of the fourth author should have been spelled A. J. Boyle.