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An Independent Investigation of the Crystal Structure of 1,8-Dinitronaphthalene (Orthorhombic Form) at 22 and 97°C

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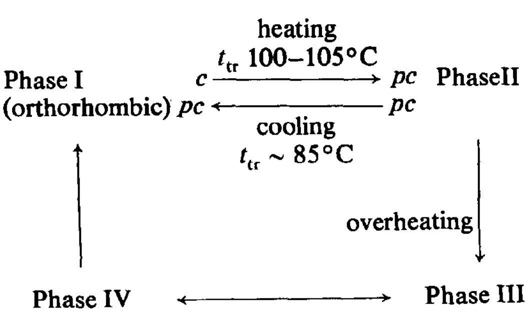
The crystal and molecular structure of the orthorhombic form of 1,8-dinitronaphthalene, $C_{10}H_6N_2O_4$, has been reinvestigated at 22°C and examined at 97°C using single-crystal diffractometer data in order to throw some light on the structural aspects of the polymorphism of the title compound. The space group of the crystal is $P2_12_12_1$ with $a = 11.375$ (*1*), $b = 14.974$ (*5*), $c = 5.388$ (*1*) Å at 22°C and $a = 11.475$ (*1*), $b = 15.002$ (*1*), $c = 5.425$ (*6*) Å at 97°C. $Z = 4$ at both temperatures. The data, collected with CuK_α radiation, were refined by full-matrix least squares to $R = 0.049$ for 1087 reflexions (22°C) and $R = 0.061$ for 1078 reflexions (97°C). The structure was found to be in agreement with that determined by Z. A. Akopian, A. I. Kitaigorodskii, T. J. Struchkov (*Zh. Strukt. Khim.* **6**, 729 (1965)). Changes occurring on heating to a temperature of 97°C, which is close to the transition temperature (of $c \rightarrow pc$ type) 100–105°C, suggest that the possible mechanism of the phase transition is that of the displacive transformation of secondary coordination. Anisotropic temperature factors of the majority of atoms increased by ca. 50% with no particular direction of atom vibrations being distinguished.

Introduction

The polymorphism of 1,8-dinitronaphthalene, henceforth called 1,8-DNN, has been studied by the DTA method (*1*), by means of the optical and X-ray techniques (*2, 3*) and the dilatometric method (*4*). The results of these studies, summarized after Jakubowski and Rohleder (*4*) below, lead to the conclusion that the title compound exists in four polymorphic modifications.

The structure of phase I of 1,8-DNN has been determined from photographic data with an isotropic approximation by Akopian et al. (*5*). The structure of phase II of 1,8-DNN cannot be found by the single-crystal X-ray technique since transformation: phase I → phase II is of the type: single crystal → polycrystalline material. The monoclinic modification of 1,8-DNN found by Kozhin (*3*) on supercooled crystals of the high-temperature form and regarded by him as phase II, may, according to Jakubowski *et al.* (*4*), correspond to phase III or IV,

The present investigation was undertaken in order to determine the structure of 1,8-DNN (phase I) more accurately at room temperature, and in particular to find out how it changes near the transition temperature of phase I to phase II.



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Collection and Treatment of Data

The crystals of the carefully purified material were grown from the solvent (90% toluene, 10% acetone) and kindly supplied by Professor J. Rohleder. The crystals are transparent flat rhombic plates.

The accurate unit-cell parameters and the intensity data were measured on an automatic four-circle diffractometer of type CAD-4

TABLE I

SUMMARY OF CRYSTAL DATA, DETAILS OF DATA COLLECTION AND STRUCTURE REFINEMENT OF THE TWO MEASUREMENTS, AT 22 AND 97°C[a]

	22°C		97°C
Crystal data			
Crystal system		Orthorhombic	
Space group		$P2_12_12_1$	
a (Å)	11.375 (1)		11.475 (1)
b (Å)	14.974 (5)		15.002 (1)
c (Å)	5.388 (1)		5.425 (6)
V (Å^3)	917.734		933.903
D_x (g cm^{-3})	1.584		1.557
D_m (g cm^{-3})	1.57		
μ(cm^{-1})		10.905	
Crystal size (mm)	0.30 x 0.28 x 0.19		0.25 x 0.25 x 0.18
Data collection			
Radiation		CuK$_\alpha$	
Monochromator		Graphite	
Instrument		Four circle diffractometer	
Scan mode		$\omega - 2\theta$	
Scan interval $\Delta\omega$	0.6 + 0.4 tan θ		0.8 + 0.4 tan θ
θ_{max}(°)		72	
Number of measured independent reflexions	1097		1124
Rejection criterion		$I_{net} < 0$	
Number of reflexions used in refinement of structure	1090		1078
Treatment of data and refinement of structure			
Lorentz and polarization corrections		Applied	
Absorption correction	For general crystal shape		Not applied
Treatment of extinction effect	Reflexions 200, 400, 040 removed		
Refinement method		Full-matrix least squares	
Weighting scheme		Applied	
Scattering factors		International Tables for X-ray Crystallography (1974)	
Anomalous dispersion correction		Not applied	
Thermal parameters		All atoms except hydrogen anisotropic	
Hydrogen atoms		Calculated positions	
Maximum electron density (e (Å^3)) on a final difference Fourier	0.28		0.22
$R = \Sigma \lvert \lvert F_0 \rvert - \lvert F_c \rvert \rvert / \Sigma \lvert F_0 \rvert$	0.049		0.061
$R_w = \Sigma(\lvert \lvert F_0 \rvert - \lvert F_c \rvert \rvert w^{1/2}) / \Sigma \lvert F_0 \rvert w^{1/2}$	0.055		0.057

[a] Where the information is identical for both, it is located in the middle column.

TABLE II

FRACTIONAL COORDINATES AND ANISOTROPIC THERMAL PARAMETERS ($\times 10^4$) AT 22°C (THE TOP ENTRY) AND AT 97°C (THE BOTTOM ENTRY) WITH THEIR STANDARD DEVIATIONS[a]

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	6558(2)	7493(1)	−1891(4)	442(10)	391(10)	506(12)	63(9)	22(10)	−76(8)
	6553(3)	7488(2)	−1897(5)	642(17)	588(15)	691(18)	80(15)	37(16)	−114(12)
O(2)	6580(2)	7917(1)	1946(5)	541(11)	364(10)	659(15)	−154(11)	−95(12)	−34(9)
	6574(3)	7913(2)	1911(6)	783(18)	522(13)	885(21)	−195(15)	−167(18)	−78(13)
O(3)	4089(3)	7557(2)	−4793(5)	588(14)	484(11)	542(13)	157(10)	−102(11)	−5(10)
	4092(3)	7553(2)	−4757(6)	839(22)	639(16)	747(19)	204(15)	−104(18)	25(15)
O(4)	4170(2)	7765(1)	−833(5)	455(11)	334(9)	601(14)	−123(10)	−62(11)	79(9)
	4173(3)	7763(2)	−826(6)	652(16)	468(14)	813(20)	−154(14)	−55(16)	90(12)
N(1)	6386(2)	7369(1)	329(5)	272(10)	276(10)	512(13)	−52(10)	−37(10)	−18(8)
	6386(2)	7361(2)	309(7)	395(14)	460(15)	661(19)	−55(15)	−61(15)	−15(12)
N(2)	4224(2)	7291(1)	−2673(5)	286(10)	322(11)	496(14)	24(10)	−46(10)	13(8)
	4223(3)	7287(2)	−2662(6)	430(14)	445(14)	668(19)	12(15)	−46(15)	26(12)
C(1)	5984(2)	6472(1)	1060(5)	287(10)	266(10)	378(13)	−45(10)	24(10)	15(9)
	5989(2)	6474(2)	1048(6)	425(15)	404(14)	511(18)	−58(15)	12(15)	28(12)
C(2)	6570(2)	6102(2)	3029(5)	358(12)	417(12)	370(13)	−24(11)	−13(11)	31(11)
	6574(3)	6103(2)	2987(7)	500(17)	602(19)	512(20)	−39(17)	−7(17)	63(15)
C(3)	6390(2)	5194(2)	3599(6)	447(13)	434(13)	425(15)	70(13)	−10(13)	105(12)
	6395(3)	5196(3)	3570(7)	636(20)	640(20)	602(22)	123(21)	−7(21)	109(19)
C(4)	5669(2)	4688(2)	2178(6)	426(12)	332(11)	478(15)	86(12)	68(13)	44(10)
	5656(3)	4693(2)	2150(7)	624(19)	501(18)	633(23)	111(19)	72(21)	73(16)
C(5)	4272(2)	4518(2)	−1274(6)	354(11)	260(10)	562(17)	−40(11)	96(13)	−50(9)
	4274(3)	4523(2)	−1254(8)	496(17)	453(16)	728(25)	−81(17)	88(21)	−56(15)
C(6)	3645(2)	4868(2)	−3206(6)	371(12)	360(12)	520(16)	−121(13)	7(13)	−62(10)
	3656(3)	4871(2)	−3176(8)	516(18)	516(18)	781(27)	−176(20)	1(21)	−85(15)
C(7)	3698(2)	5789(2)	−3681(6)	333(11)	356(11)	433(14)	−43(11)	−5(12)	−4(10)
	3699(3)	5797(2)	−3667(8)	465(16)	536(18)	591(22)	−70(17)	−1(18)	−22(15)
C(8)	4397(2)	6327(1)	−2288(5)	279(10)	287(10)	375(13)	−36(10)	39(10)	8(9)
	4386(2)	6331(2)	−2248(6)	371(13)	408(15)	534(18)	−18(15)	37(15)	27(12)
C(9)	5144(2)	6000(1)	−367(5)	297(10)	221(9)	346(12)	−24(9)	70(10)	20(8)
	5138(3)	5999(2)	−373(6)	430(15)	402(14)	437(16)	−27(14)	53(15)	−1(12)
C(10)	5034(2)	5070(1)	165(5)	354(11)	239(10)	418(15)	−8(11)	67(12)	7(9)
	5022(2)	5070(2)	175(7)	471(16)	384(15)	578(21)	−32(16)	109(17)	22(13)
H(1)	7163	6502	4132						
	7170	6501	4067						
H(2)	6829	4900	5177						
	6837	4900	5125						
H(3)	5574	3985	2578						
	5556	3992	2551						
H(4)	4190	3817	−830						
	4193	3824	−817						
H(5)	3112	4439	−4360						
	3134	4439	−4324						
H(6)	3179	6072	−5165						
	3192	6078	−5152						

[a] The anisotropic thermal parameters of nonhydrogen atoms are in the form. $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}hla^*c^* + 2U_{12}hka^*b^*)]$. Isotropic thermal parameter of the hydrogen atoms $u = 0.05$ Å^2.

(manufactured by Enraf–Nonius, Delft, Holland) equipped with an apparatus enabling one to heat the crystal with a continuous stream of hot nitrogen gas. The temperature of the crystal during the experiment was 97 ± 2°C. The data were collected on two different crystals at two temperatures. For measurement at 97°C the crystal had to be sealed in a capillary tube.

Some of the details of the two different crystals, the method used to collect the data and refine the structure are given in Table I.

During the data collection the scan speed was calculated from the net intensity in a fast prescan. The scan was continuous and the background intensity was measured for $\frac{1}{4}$ of the scan time at both ends of the scan interval. Two control reflexions were measured after each block of 25 reflexions to check for crystal decomposition and radiation stability. Maximum intensity variation over the data collection period of 50 and 60 hr were 3.5 and 4.6% for measurements at 22 and 97°C, respectively. All intensities and their variances were scaled according to these control reflexions.

The refinement was undertaken with the atom coordinates found by Akopian *et al.* (*5*). It was carried out using programs written by Sheldrick (*6*) and went smoothly for the data collected at both temperatures. The function minimized was $\Sigma w|F_0 - kF_c|^2$ with weights $w = (w'\,(2F_{\min} + F_0 + 2F_0^2/F_{\max}))^{-1}$, where w' is statistical weights. The intensities collected at 22°C were corrected for an absorption effect with the program DATAPC which is a modification by Svensson (*7*) of a program originally written by Coppens *et al.* (*8*). Since this correction had a minor effect on the quality of the data it was felt that the labor involved in applying the correction to the intensities collected at 97°C was not justified. The positions of hydrogen atoms were calculated assuming a C–H bond distance of 1.08 Å and most of them were in good agreement with those revealed on a difference Fourier map. Theoretical positions of hydrogen atoms with an assigned isotropic temperature factor U of 0.05 Å^2 were included in the calculations of the structure factors but not refined because of the limited number of independent reflexions. The positions of the hydrogens were recalculated after the other atoms had shifted in response to their inclusion. Refinement was continued with the atoms vibrating anisotropically until the final average ratio of shift to standard deviation was 0.05 and 0.01, and the maximum ratio was 0.1 and 0.04 at 22 and 97°C, respectively.

Table II lists the final coordinates and coefficients of the anisotropic temperature factors with their standard deviations. Tables III and IV[1] list the observed structure amplitudes and the calculated structure factors for the experiments at 22 and 97°C, respectively.

TABLE V

	Akopian *et al.* (*5*) Room temperature	← Δ →	Present work 22°C	← Δ →	97°C
a (Å)	11.352 (*2*)	0.020 (*2*)	11.375 (*1*)	0.100 (*2*)	11.475 (*1*)
b (Å)	14.934 (*2*)	0.040 (*5*)	14.974 (*5*)	0.028 (*5*)	15.002 (*1*)
c (Å)	5.376 (*1*)	0.012 (*1*)	5.388 (*1*)	0.037 (*6*)	5.425 (*6*)

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[1] See NAPS document no. 03033 for 11 pages of supplementary material. Order from ASIS/NAPS c/o Microfiche Publications, P. O. Box 3513, Grand Central Station, New York, New York 10017. Remit in advance for each NAPS Accession number. Institutions and organizations may use purchase orders when ordering, however, there is a billing charge for this service. Make checks payable to Microfiche Publications. Photocopies are $5.00. Microfiches are $3.00. Outside of the U.S. and Canada, postage is $3.00 for a photocopy or $1.50 for a fiche.

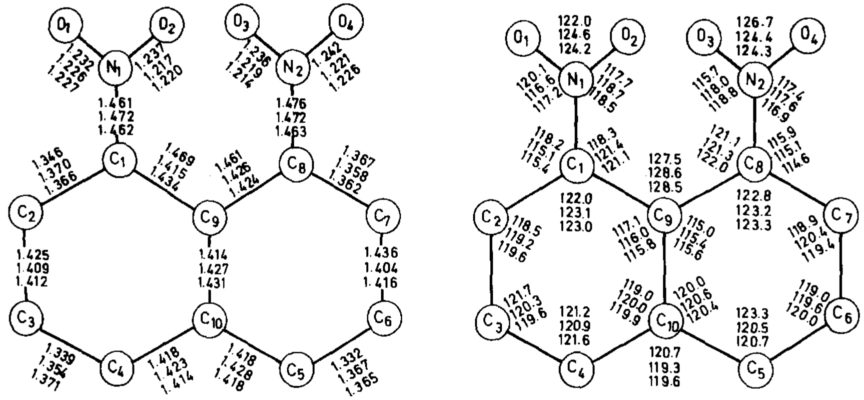

FIG. 1. Bond lengths (Å) and bond angles (°) as determined by Akopian *et al.* (*5*) (the top line), determined in the present study at 22°C (the middle line) and at 97°C (the bottom line). The estimated standard deviations for the latter two are 0.003–0.005 Å in distances and 0.2–0.4° in angles.

Discussion

The crystal and molecular structure of 1,8-dinitronaphthalene, determined from 915 reflexions collected photographically at room temperature, was described and discussed in detail by Akopian *et al.* (*5*). The results of the present investigation confirm it, although the

TABLE VI

SOME SELECTED NONBONDED DISTANCES (Å) AND ANGLES (°)

	22°C	97°C	Δ		22°C	97°C	Δ
C3–C6	3.599	3.638	0.039	C1–C4	2.762	2.765	0.003
C2–C7	3.747	3.793	0.046	C8–C5	2.767	2.768	0.001
$O1^b$–O3	3.390	3.433	0.043	$O4^b$–N1	3.185	3.215	0.030
$O1^b$–O4	3.336	3.371	0.035	$O4^b$–C1	3.801	3.830	0.029
$O1^b$–N2	3.918	3.952	0.034	$O4^c$–C3	3.883	3.905	0.023
$O1^c$–C5	3.326	3.351	0.025	$O4^c$–C4	3.493	3.522	0.029
$O1^c$–C6	3.565	3.583	0.021	$O4^c$–C5	3.529	3.558	0.029
$O2^b$–O3	3.317	3.349	0.032	$N1^b$–O3	3.906	3.934	0.028
$O2^b$–O4	3.175	3.204	0.029	$N1^b$–O4	3.185	3.215	0.030
$O2^b$–N2	3.049	3.082	0.033	$N1^b$–N2	3.504	3.536	0.032
$O2^b$–C7	3.229	3.256	0.027	$N1^c$–C4	3.960	3.992	0.032
$O2^b$–C8	3.404	3.425	0.021	$N1^c$–C6	3.913	3.940	0.027
$O2^c$–C4	3.715	3.734	0.019	$N2^b$–O2	3.049	3.082	0.033
$O2^c$–C5	3.482	3.512	0.030	$N2^b$–O1	3.918	3.952	0.034
$O2^c$–C6	3.559	3.578	0.019	$N2^b$–N1	3.504	3.536	0.032
$O3^b$–O1	3.390	3.433	0.043	$N2^b$–C2	3.866	3.887	0.021
$O3^b$–O2	3.317	3.349	0.032	$N2^c$–C5	3.790	3.818	0.028
$O3^b$–N1	3.906	3.934	0.028	$C2^a$–C3	3.853	3.869	0.016
$O3^b$–N2	3.626	3.651	0.025	$C2^b$–O3	3.626	3.651	0.025
$O3^c$–C4	3.452	3.475	0.023	$C2^b$–O4	3.608	3.629	0.021
$O3^c$–C5	3.525	3.543	0.018	$C3^a$–C4	3.865	3.905	0.040
$O3^c$–C10	3.898	3.918	0.020	$C5^a$–C6	3.819	3.863	0.044
$O4^b$–O1	3.336	3.371	0.035	$C5^a$–C7	3.684	3.721	0.037
$O4^b$–O2	3.175	3.204	0.029	$C6^a$–C7	3.744	3.781	0.037

Note: Superscripts refer to atoms in the following positions: a, $1.5 - x$, $1 - y$, $z - 0.5$; b, $x - 0.5$, $1.5 - y$, $1 - z$; c, $1 - x, y - 0.5$, $1.5 - z$. Atoms without superscripts are in positions x, y, z as given in Table II.

comparison of the unit-cell parameters (Table V) shows that the differences are greater than three times their e.s.d.

If the same criterion of the significance of the difference is applied to the molecular bond lengths and angles given in Fig. 1, it can be seen that the four bond lengths (C6–C7, C8–C9, C1–C2, C6–C5), and the six bond angles involving nitro groups differ from each other. These differences may be accounted for by the different techniques of data collection in both studies and by the differences in the structure refinement, mainly the application of an isotropic (Akopian *et al.* (*5*)) and an anisotropic (present work) approximation. They do not change, however, the fact that both refinements lead to basically the same structure.

For the study of the polymorphic changes of 1,8-dinitronaphthalene it is interesting to compare the structures determined at 22 and 97°C. The most obvious difference is apparent in the values of the anisotropic temperature factors, which for the majority of atoms increased by ca 50% (Table II), with no particular direction of atomic vibrations being distinguished.

Figure 1 shows the bond lengths and bond angles found for the molecule at both tem-

TABLE VII

DEVIATIONS OF THE ATOMS FROM THE LEAST-SQUARES PLANES (Å) AND ANGLES (°) BETWEEN THESE PLANES[a]

	22°C	97°C
Naphthalene plane I	$-8.4759X + 3.0535Y + 3.4212Z = -2.6526$	$-8.5055X + 3.0784Y + 3.4672Z = -2.6523$
Benzene rings		
plane II	$-8.5014X + 3.5111Y + 3.3495Z = -2.4282$	$-8.4873X + 3.6140Y + 3.4091Z = -2.3560$
plane III	$-8.5108X + 2.5638Y + 3.4537Z = -2.9362$	$-8.5919X + 2.5300Y + 3.4777Z = -2.9850$
Nitro group		
N10102 plane IV	$10.5828X - 5.2934Y + 0.5243Z = 2.8747$	$10.7230X - 5.1478Y + 0.5154Z = 3.0743$
N20304 plane V	$11.1598X + 2.7100Y - 0.3706Z = 6.7888$	$11.2713X + 2.6195Y - 0.3722Z = 6.7678$

	Plane I	II	III		Plane I	II	III
C1	-0.0806^x	-0.0316^x	-0.1313	C1	-0.0853^x	-0.0300^x	-0.1583
C2	-0.0166^x	-0.0002^x	-0.0449	C2	-0.0248^x	0.0004^x	-0.0804
C3	0.0537^x	0.0249^x	0.0724	C3	0.0504^x	0.0233^x	0.0466
C4	0.0242^x	-0.0158^x	0.0655	C4	0.0317^x	-0.0154^x	0.0605
C5	-0.0243^x	-0.0437	0.0190^x	C5	-0.0254^x	-0.0643	0.0211^x
C6	-0.0473^x	-0.0352	-0.0252^x	C6	-0.0590^x	-0.0693	-0.0283^x
C7	0.0265^x	0.0840	0.0018^x	C7	0.0192^x	0.0615	-0.0017^x
C8	0.0749^x	0.1452	0.0259^x	C8	0.0913^x	0.1551	0.0366^x
C9	-0.0009^x	0.0388^x	-0.0302^x	C9	-0.0004^x	0.0361^x	-0.0414^x
C10	-0.0096^x	-0.0160^x	0.0087^x	C10	0.0023^x	-0.0143^x	0.0138^x
N1	-0.3975	-0.3033	-0.4959	N1	-0.4062	-0.2984	-0.5319
N2	0.3841	0.5018	0.2873	N2	0.3807	0.4979	0.2745
O1	-1.2649	-1.1496		O1	-1.2740	-1.1463	
O2	0.1586	0.2658		O2	0.1593	0.2877	
O3	-0.1455		-0.2618	O3	-1.1524		-0.2742
O4	1.2042		1.0903	O4	1.2063		1.0764

Angle between planes	22°C	97°C	Δ
$\widehat{\mathrm{I\ \ IV}}$	45.29°	45.38°	0.09°
$\widehat{\mathrm{I\ \ V}}$	42.45°	42.60°	0.15°
$\widehat{\mathrm{IV\ \ V}}$	147.36°	148.37°	1.01°
$\widehat{\mathrm{II\ \ III}}$	176.21°	175.76°	0.45°

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[a] Coordinates X, Y, Z refer to crystal axes a, b, c.

[b] Superscript x refers to atoms for which least-squares plane was calculated.

peratures. With the exception of the C1–C9 bond, the differences are smaller than three times the e.s.d. However, the change in the nonbonded distances C2–C7 and C3–C6 (Table VI), which lie approximately in the (010) plane, is significant, whereas the perpendicular distances C1–C4 and C8–C5 (Table VI) remain practically unchanged. That could be explained as being the result of the thermal agitation of atoms, due to which the midpoints of the atom vibrations shift from their original positions. This leads to small changes in the planarity of naphthalene (Table VII), and thus in the distances close in direction to (010) plane, leaving at the same time the aromatic plane practically rigid in [010] direction.

It was suspected that on heating the angles of twist of the nitro groups out of the naphthalene plane might change, or that some disorder might appear, preceding the phase transition. However, neither the difference Fourier map nor the analysis of angles between least-squares planes (Table VII) shows these changes present. This is understandable since packing of the molecules, shown in Fig. 2, did not change with temperature sufficiently to weaken the van der Waals interactions of nitro groups with neighboring carbon and hydrogen atoms. The details of the molecular packing were discussed by Akopian *et al.* (*5*). In the present work only the differences occurring on heating are given.

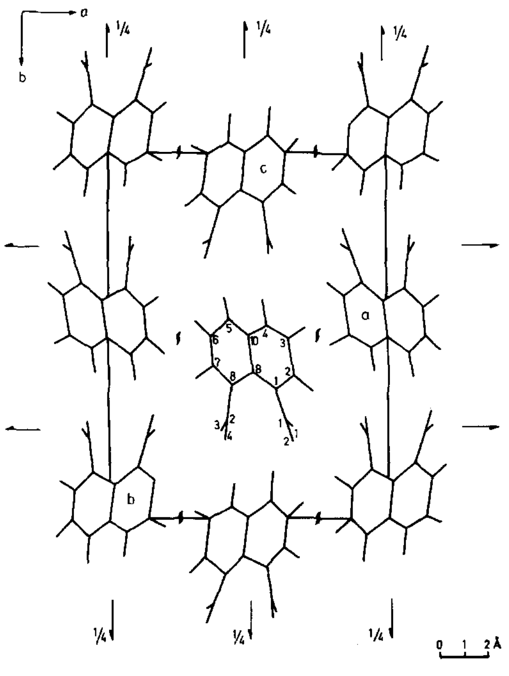

FIG. 2. Projection of the structure along [001], after Akopian *et al.* (*5*).

And thus, the inspection of the intermolecular distances (Table III), shows that a number of them change with an increase in temperature of more than 0.02 Å, which is three times the e.s.d., and that the closer their direction is to the (010) plane the stronger they are affected by the change in temperature.

The obtained results enable the interpretation of the anisotropic thermal expansion, found in the dilatometric study (*4*), in terms of the changes occurring on a molecular level. As a consequence the unit-cell parameters change as given in Table V: period *a* is most affected by the change in temperature and period *b* least affected.

On the basis of these observations one may be tempted to suggest that the mechanism of the polymorphic change, in terms of Burger's structural classification of polymorphic transformations (*9*), can be described as the displacive transformation of secondary coordination. To support this conclusion it should be noticed that the specific volume changes from 0.631 cm^3/g at 22°C to 0.642 cm^3/g at 97°C, leading to the more open high-temperature form. The high-temperature monoclinic form reported by Kozhin (*3*) would have specific volume 0.656 cm^3/g.

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