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The Crystal Structure of the Free Radical Tri-*p*-nitrophenylmethyl

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An X-ray crystallographic investigation of the free radical tri-*p*-nitrophenylmethyl has been carried out. The crystals belong to the orthorhombic system, space group *Pbcn*. The unit cell containing four molecules has the following dimensions: a = 7.80 Å, b = 11.50 Å, c = 18.60 Å. The crystal structure has been roughly determined in projections along the a and b axes by Fourier methods, and refined three-dimensionally by least squares procedures to an R value of 11.1 %. The temperature motion was assumed to be isotropic for all atoms except the oxygens. The space group requires a two-fold symmetry of the molecule, which is propeller-shaped but does not have a trigonal symmetry. The motion of the nitro groups is highly anisotropic or the groups are possibly disordered. The central C—C bond length is 1.47 Å and all other distances are normal within the accuracy.

The crystal structure investigation of the tri-*p*-nitrophenylmethyl free radical was carried out to compare the results with those obtained in a gas electron diffraction investigation of triphenylmethyl.[1] The electron diffraction investigation did not give an unequivocal answer to the question of whether the central C—C bonds were coplanar or not, since the apparent deviation could be due to heavy thermal motion. One could expect to obtain more accurate informations about the angular parameters, both the central angles and the twist angles of the phenyl groups, from a crystal structure determination. There is a possibility, however, that the angular parameters may be influenced by the packing of molecules in the crystal, and these results may therefore be different from what is determined in the gaseous state. Structural differences of this kind have been observed, for instance, in the gaseous electron diffraction and X-ray crystallographic investigation of biphenyl.[2–4]

The free radical tri-*p*-nitrophenylmethyl has been known for years to be stable in the crystalline state. Gomberg[5] prepared the radical in solution without isolating it. He noticed the strong intensity of the colour of a solution of tri-*p*-nitrophenylmethyl chloride when silver was added, and he assumed that the methyl derivative was very little associated. The first synthesis and isolation of the tri-*p*-nitrophenylmethyl radical was carried out by Ziegler and Boye.[6] Even if nothing was said explicitly, Ziegler and Boye seemed to have been of the opinion that the radical was present in the solid state. In 1936 Allen and Segden [7] measured the paramagnetism of tri-*p*-nitrophenylmethyl and concluded that the crystals largely consisted of the free radical.

EXPERIMENTAL

Attempts to grow crystals suitable for X-ray work of tri-*p*-nitrophenylmethyl from a sample prepared according to Ziegler and Boye did not succeed owing to the insolubility of the radical. The difficulty was overcome by slightly changing the procedure. By keeping a zinc rod in a solution of tri-*p*-nitrophenylmethyl bromide in a mixture of benzene and acetone, fairly well developed crystals of the tri-*p*-nitrophenylmethyl radical were formed on the zinc rod surface. The presence of the radical was controlled by electron spin resonance.

CRYSTAL AND INTENSITY DATA

The tri-*p*-nitrophenylmethyl crystals decomposed gradually and were kept in Lindemann glass tubes during the X-ray exposures, all made at room temperature. The crystals are orthorhombic and the unit cell dimensions determined from Weissenberg photographs (Cu$K\alpha$ radiation) along [100] and [010] are:

$$a = 7.80 \text{ Å}, \ b = 11.50 \text{ Å}, \ c = 18.60 \text{ Å}$$

The density determined by flotation is 1.5_0 g/cm^3 and the calculated density assuming 4 molecules in the unit cell is 1.50_5 g/cm^3. Integrated equi-inclination Weissenberg photographs of $h = 0$ through 4 and $k = 0$ were collected and the intensities measured on a microphotometer. The weak intensities were estimated visually. The intensity determination is probably not as reliable as usual, owing to the decomposition of the crystal during the exposure. No absorption correction was applied.

According to the systematic absences the space group is *Pbcn*, and with four molecules in the cell there is a two-fold symmetry requirement to the molecule. However, a limited number of *hk*0 reflections were observed, and there could be some doubt as to whether the extinction rule $h + k \neq 2n$ was present or not. During the preliminary work in projections the space group was erroneously assumed to be $Pca2_1$[8] (a and b axis interchanged). The refinement proceeded to a lower R value by ignoring the symmetry requirement to the molecule. The refinement of the three-dimensional data showed, however, that *Pbcn* is the correct space group.

STRUCTURE DETERMINATION *

The structure was initially solved in projections. The [100] Patterson projection revealed the three-fold symmetry of the molecule, and a Fourier

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* With the exception of programs listed in footnote all programming has been carried out at this institute.

projection was successfully refined to an *R* value of 13 % using isotropic damping. This refinement resulted in a slight deviation from the symmetry requirement of *Pbcn*. Because of heavy overlapping the [010] projection could not be satisfactorily refined.

The intensity data were brought to a common scale by means of the *h0l* intensities. The 202 reflection which was too strong to be properly normalized from the *2kl* data was included from *h0l*. Of the 262 observed reflections 49 were visually estimated. Few reflections were observed outside $\sin \theta = 0.5$ and non-observable reflections were included only if $\sin \theta$ was less than 0.5 and assigned a value equal to half the smallest observable ($I_{\min}$). A total of 316 reflections were used in the three-dimensional refinement. By successive block diagonal least squares refinement, using isotropic temperature factors, and allowing scale factors for the different layers to be refined, the *R* value decreased from 32 % to 15 %. The hydrogens were included in fixed positions. All parameters were not refined simultaneously owing to the large number of independent parameters compared with the number of reflections. At this stage least squares planes through the central carbon atom, the phenyl groups, and the nitrogen atoms were calculated. It was clearly revealed that the plane normals fulfilled the two-fold symmetry requirement of the space group *Pbcn*, which was then considered the correct one.

The unobserved reflections were then assigned a value equal to 0.25 $I_{\min}$. Further refinement on the reduced number of independent parameters in space group *Pbcn* proceeded easily to an *R* value of about 15 %. A full matrix least squares refinement * after adjusting the hydrogen positions and omitting the strong 202 reflection, resulted in a very high isotropic thermal damping of the oxygens. Neither a total three-dimensional difference Fourier synthesis nor a difference synthesis ** with only the oxygen contribution removed indicated whether highly anisotropic vibrations were present or a statistical distribution. A least squares refinement on the microphotometered reflections only and isotropic temperature factors resulted in an *R* value of 8.7 %, while the corresponding *R* value for all data was about 13 %. An adjustment of the weighting factor for the visually observed reflections and introduction of anisotropic temperature factors for the oxygens reduced the *R* index to 11.1 % for all reflections. The observed and calculated structure factors are listed in Table 1. The final coordinates, thermal parameters and their standard deviations are given in Table 2. The standard deviations are probably somewhat underestimated, as the visually observed and unobserved reflections have been assigned weights which are too low.

The authors believe that the determined intensities do not have the usual accuracy owing to the decomposition of the crystal during the X-ray exposure, and that a further refinement introducing more parameters is not advisable. The errors introduced due to decomposition may well be systematic.

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* Program written by P. K. Gantzel, R. A. Sparks, and K. N. Trueblood (*IUCr World List* No. 384), modified and adapted for UNIVAC 1107 by C. Rømming.

** Program written by P. K. Gantzel and H. Hope, adapted for UNIVAC 1107 by H. Hope.

Table 1. Observed and calculated structure factors. The five columns are *h*, *k*, *l*, $10F_0$, and $10F_c$. Visually estimated reflections are marked V. Non-observed reflections, marked *, are included with the values used in the least squares refinement.

0	0	2	411	-399	
0	0	4	1045	1061	
0	0	6	603	529	
0	0	8	283	-275	
0	0	10	242	275	
0	0	12	322	320	
0	0	14	167	-172	
0	0	16	465	525	
0	0	18	129	-181	V
0	0	20	117	123	V
0	2	0	820	847	
0	2	1	435	-387	
0	2	2	277	-291	
0	2	3	481	416	
0	2	4	615	-593	
0	2	5	696	-741	
0	2	6	468	-459	
0	2	7	28	-17	*
0	2	8	624	-632	
0	2	9	605	-594	
0	2	10	448	-456	
0	2	11	35	-101	*
0	2	13	134	161	
0	2	15	168	-163	
0	2	17	130	-199	V
0	2	19	127	126	V
0	4	0	890	-832	
0	4	1	59	-32	V
0	4	2	197	179	
0	4	3	304	-283	
0	4	4	78	28	V
0	4	5	28	-32	*
0	4	6	28	-6	*
0	4	7	123	101	
0	4	8	96	-78	V
0	4	9	102	-3	V
0	4	10	108	-135	V
0	4	11	113	-66	V
0	4	12	203	222	
0	4	13	122	52	V
0	4	19	146	133	
0	6	0	966	-974	
0	6	1	236	247	
0	6	2	31	-10	*
0	6	3	480	-515	
0	6	4	126	-101	
0	6	5	35	10	*
0	6	6	145	-171	
0	6	7	86	91	V
0	6	8	111	58	V
0	6	10	334	341	
0	6	13	133	-113	V
0	8	0	114	87	V
0	8	1	191	-190	
0	8	2	147	105	
0	8	3	84	-30	V
0	8	4	217	200	
0	8	5	248	221	
0	8	7	413	-436	
0	8	8	326	337	
0	8	9	620	666	
0	8	10	210	226	
0	8	11	130	-85	V
0	10	5	130	-91	V
0	10	7	247	-231	
0	10	10	160	-127	
1	0	2	395	-352	
1	0	4	518	-464	
1	0	6	840	888	
1	0	8	318	332	
1	0	10	403	374	
1	0	12	385	443	
1	1	0	465	465	
1	1	1	218	-215	
1	1	2	473	442	
1	1	3	498	468	
1	1	4	784	-800	
1	1	5	26	59	*
1	1	6	134	171	
1	1	7	275	-270	
1	1	8	36	-28	*
1	1	9	212	-203	
1	1	10	39	-40	*
1	1	11	42	33	*
1	1	12	217	219	
1	1	13	192	-166	
1	1	17	281	-299	
1	2	1	840	837	
1	2	2	633	-585	
1	2	3	1045	1070	
1	2	4	267	-257	
1	2	5	515	487	
1	2	6	223	-204	
1	2	7	160	-149	
1	2	8	35	15	*
1	2	9	180	145	
1	2	10	41	75	*
1	2	11	42	63	*
1	2	16	175	-180	
1	3	0	270	277	
1	3	1	465	429	
1	3	2	651	-629	
1	3	3	241	241	
1	3	4	36	36	*
1	3	5	338	352	
1	3	6	450	400	
1	3	7	203	189	
1	3	8	724	-712	
1	3	9	42	108	*
1	3	10	202	200	
1	3	12	141	-157	
1	4	1	176	-140	
1	4	2	107	-128	
1	4	3	253	278	
1	4	4	86	24	V
1	4	5	137	-166	
1	4	6	412	-392	
1	4	7	263	-259	
1	4	8	210	193	
1	4	9	267	-248	
1	4	10	185	206	
1	4	11	191	-164	
1	5	0	132	-108	
1	5	1	748	-725	
1	5	2	96	66	V
1	5	3	321	308	
1	5	4	357	354	
1	5	5	39	-81	*
1	5	6	40	42	*
1	5	7	42	81	*
1	5	8	42	-25	*
1	5	9	135	130	
1	5	11	275	-267	
1	6	1	318	-301	
1	6	2	102	-30	V
1	6	3	136	121	
1	6	4	121	-123	
1	6	5	151	-131	
1	6	6	192	-206	
1	6	7	157	155	
1	6	10	156	40	
1	7	0	163	-199	
1	7	1	171	-151	
1	7	2	157	123	
1	7	3	44	-39	*
1	7	10	216	210	
1	8	1	181	-178	
1	8	3	243	-231	
1	8	7	176	187	
1	9	5	168	-115	
1	9	6	220	-195	
1	9	8	251	274	
1	9	11	222	230	
1	10	7	202	208	
2	0	0	884	928	
2	0	2	2125	-2580	
2	0	4	33	46	*
2	0	6	41	-116	*
2	0	8	49	-109	*
2	0	10	56	112	*
2	0	14	605	-668	
2	1	1	494	545	
2	1	2	1032	-1107	
2	1	3	498	550	
2	1	4	162	164	
2	1	5	207	164	
2	1	6	428	-422	
2	1	7	246	260	
2	1	8	119	44	V
2	1	9	50	99	*
2	1	10	52	91	*
2	1	11	284	269	
2	1	14	269	244	
2	2	0	147	-160	
2	2	1	409	364	
2	2	2	382	-357	
2	2	3	815	882	
2	2	4	213	232	
2	2	5	379	-414	
2	2	6	483	494	
2	2	7	354	354	
2	2	8	216	222	
2	2	9	138	-118	V
2	2	10	60	42	*
2	2	11	166	137	V
2	3	1	423	-431	
2	3	2	179	176	
2	3	3	129	127	
2	3	4	338	-308	
2	3	5	513	-540	
2	3	6	47	96	*
2	3	7	304	-284	
2	3	8	199	-220	
2	3	9	60	-125	*
2	3	10	62	49	*
2	4	0	103	-61	V
2	4	1	274	247	
2	4	2	186	194	
2	4	3	199	185	
2	4	4	265	226	
2	4	5	49	14	*
2	4	6	332	357	
2	4	7	52	94	*
2	4	8	57	74	*
2	4	9	62	59	*
2	5	1	50	108	*
2	5	2	328	329	
2	5	3	138	36	V
2	5	4	194	186	
2	5	5	248	251	
2	5	6	56	136	*
2	5	7	201	-180	
2	6	0	174	-195	
2	6	1	204	180	
2	6	2	330	292	
2	6	3	188	-161	
2	6	4	172	-91	V
2	6	5	60	176	*
2	6	12	286	-300	
2	8	5	328	347	
2	8	6	313	-313	
2	8	7	433	-432	
2	8	9	259	218	
2	8	11	267	-236	
2	9	7	304	299	
3	0	2	362	383	
3	0	4	541	-620	
3	0	6	377	395	
3	0	8	56	-136	*
3	1	0	181	-202	
3	1	1	72	-112	V
3	1	2	106	-90	
3	1	3	352	362	
3	1	4	152	-132	
3	1	5	288	-282	
3	1	6	298	266	
3	1	7	146	-137	V
3	1	8	180	-174	V
3	1	9	263	202	
3	1	11	184	153	V
3	1	14	327	-290	
3	1	15	275	212	
3	2	1	98	-131	V
3	2	2	108	-104	V
3	2	3	106	-125	V
3	2	4	318	297	
3	2	5	352	-390	
3	2	6	250	-242	
3	2	7	220	-227	
3	2	8	186	-103	V
3	2	9	220	200	
3	3	0	110	85	V
3	3	1	206	177	
3	3	2	106	85	V
3	3	3	148	-124	V
3	3	4	148	166	V
3	3	5	528	-606	
3	3	6	533	509	
3	3	7	352	-375	
3	3	8	66	-39	*
3	4	1	52	-52	*
3	4	2	126	86	V
3	4	3	284	282	
3	4	4	548	554	
3	4	5	260	-241	
3	4	6	250	-203	
3	4	7	68	55	*
3	5	0	178	-175	V
3	5	1	214	-163	
3	5	2	62	-41	*
3	5	3	62	75	*
3	5	4	62	-111	*
3	5	5	268	-255	
3	6	1	164	28	V
3	6	3	424	482	
3	6	5	181	-178	V
4	0	0	530	549	
4	0	2	336	-375	
4	0	4	109	110	V
4	0	6	317	-339	
4	0	8	195	195	
4	0	12	426	444	
4	0	14	225	-230	V
4	1	1	30	1	*
4	1	2	36	-32	*
4	1	3	340	-380	
4	1	4	507	538	
4	1	5	219	-249	
4	1	6	201	-173	
4	1	7	67	-49	*
4	1	8	236	203	
4	1	12	397	-368	
4	2	0	279	264	
4	2	1	120	123	
4	2	2	50	58	*
4	2	3	52	11	*
4	2	4	52	91	*
4	2	5	377	-419	
4	2	6	63	21	*
4	2	7	213	190	
4	2	11	294	269	
4	3	1	182	142	
4	3	2	242	229	
4	3	3	113	65	V
4	3	4	120	4	V
4	3	5	222	-205	
4	3	6	210	126	
4	3	7	169	134	V
4	3	8	150	-176	V
4	4	0	59	-14	*
4	4	1	59	-5	*
4	4	2	215	-232	
4	4	3	206	-178	
4	4	4	161	-96	V
4	4	5	277	-276	
4	6	2	303	335	

RESULTS AND DISCUSSION

Bond distances, angles, and standard deviations calculated from the parameters from the least squares refinement are listed in Table 3. Distances and angles are also indicated in Fig. 1 where the numbering of atoms used in Table 2 is shown. If all distances are considered independently measured the mean value of the ring bond lengths is normal and equal to 1.38_5 Å. The mean values of the C—N and N—O bonds are 1.51_5 Å and 1.19_7 Å, respectively. These mean values are not significantly different from those found by Trotter [9] for nitrobenzene and Abrahams [10] for *p*-dinitrobenzene. The average central carbon-carbon bond length of 1.47 Å agrees very well with the value 1.48 Å found in the gas electron diffraction investigation of triphenylmethyl [1] and is 0.06 Å shorter than the central C—C bond in triphenylmethane.[11]

The molecules are fairly effectively packed. The packing density specified by Kitaigorodskii [12] has been calculated to be 0.70_3. The shortest intermolec-

Table 2. Positional and thermal parameters from the least squares refinement (in fractional coordinates and Å^2) and their e.s.d.'s. For the oxygen atoms the isotropic temperature factor in parenthesis are from a parameter set with $R = 12.8\ \%$. The anisotropic thermal parameters ($\times\ 10^4$) are given below the assumed parameters for the hydrogen atoms. The anisotropic temperature factor is given by

$$\exp-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl).$$

Atom	x	y	z	B
C_0	0.0000	0.1002	0.2500	3.15
		21		71
C_1	0.0000	0.2291	0.2500	3.18
		22		67
C_2	0.0503	0.2893	0.3107	3.15
	24	14	10	45
C_3	0.0581	0.4113	0.3106	3.62
	24	15	10	50
C_4	0.0000	0.4683	0.2500	3.18
		21		67
C_1'	−0.0573	0.0408	0.3148	3.00
	24	14	9	47
C_2'	−0.1725	0.0869	0.3638	3.88
	26	16	9	47
C_3'	−0.2260	0.0262	0.4242	3.15
	25	16	9	43
C_4'	−0.1521	−0.0777	0.4377	3.33
	26	17	9	44
C_5'	−0.0384	−0.1296	0.3942	2.92
	25	14	9	43
C_6'	0.0134	−0.0721	0.3316	2.80
	26	14	9	43
N_1	0.0000	0.6012	0.2500	4.70
		23		80
N_1'	−0.2181	−0.1373	0.5040	4.54
	29	17	9	47
O_1	0.0915	0.6398	0.2956	(5.33
	18	10	7	56)
O_1'	−0.3062	−0.0858	0.5440	(8.97
	25	13	9	61)
O_2'	−0.1481	−0.2307	0.5152	(6.40
	22	14	7	51)
H_2	0.0780	0.2481	0.3593	3.50
H_3	0.1123	0.4520	0.3570	3.50
H_2'	−0.2114	0.1703	0.3587	3.50
H_3'	−0.3327	0.0540	0.4568	3.50
H_5'	−0.0050	−0.2185	0.4053	3.50
H_6'	0.1227	−0.0918	0.2985	3.50

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O_1	262	94	36	−201	66	−30
	41	14	5	44	26	16
O_1'	595	133	67	61	255	−33
	72	18	8	64	43	21
O_2'	400	122	35	10	22	15
	52	16	6	56	31	17

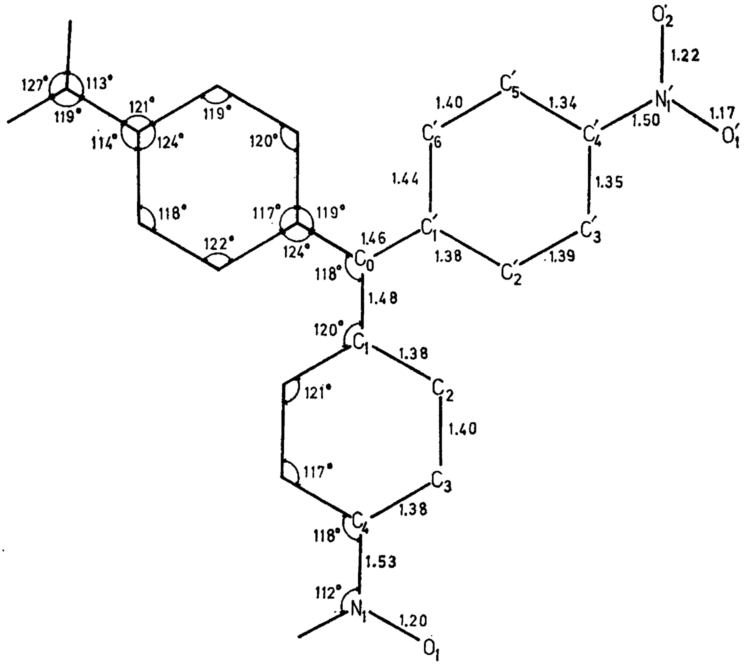

Fig. 1. Numbering of atoms, bond distances (Å), and angles in tri-*p*-nitrophenylmethyl.

Table 3. Bond distances, angles and their standard deviations in tri-*p*-nitrophenylmethyl.

	(Å)		(°)
$C_0 - C_1$	1.483 (35)	$C_1 - C_0 - C_1'$	118.0 (1.1)
$C_0 - C_1'$	1.457 (21)	$C_0 - C_1 - C_2$	120.1 (1.1)
$C_1 - C_2$	1.381 (22)	$C_0 - C_1' - C_2'$	124.3 (1.6)
$C_2 - C_3$	1.404 (23)	$C_0 - C_1' - C_6'$	118.9 (1.6)
$C_3 - C_4$	1.380 (21)	$C_1 - C_2 - C_3$	120.8 (1.7)
$C_1' - C_2'$	1.384 (26)	$C_2 - C_3 - C_4$	117.5 (1.7)
$C_2' - C_3'$	1.388 (25)	$C_1' - C_2' - C_3'$	122.3 (1.7)
$C_3' - C_4'$	1.350 (27)	$C_2' - C_3' - C_4'$	117.9 (1.7)
$C_4' - C_5'$	1.341 (26)	$C_3' - C_4' - C_5'$	124.3 (1.7)
$C_5' - C_6'$	1.399 (23)	$C_4' - C_5' - C_6'$	118.9 (1.6)
$C_6' - C_1'$	1.444 (24)	$C_5' - C_6' - C_1'$	119.6 (1.6)
$C_4 - N_1$	1.528 (35)	$C_6' - C_1' - C_2'$	116.8 (1.6)
$C_4' - N_1'$	1.501 (26)	$C_3 - C_4 - N_1$	118.4 (1.1)
$N_1 - O_1$	1.195 (16)	$C_3' - C_4' - N_1'$	114.2 (1.7)
$N_1' - O_1'$	1.174 (26)	$C_5' - C_4' - N_1'$	121.3 (1.7)
$N_1' - O_2'$	1.223 (26)	$C_4 - N_1 - O_1$	111.8 (1.3)
		$C_4' - N_1' - O_1'$	119.4 (1.8)
		$C_4' - N_1' - O_2'$	112.8 (1.8)
		$O_1' - N_1' - O_2'$	126.7 (1.8)

Table 4. Shortest intermolecular distances in the tri-*p*-nitrophenylmethyl structure.

	Å		Å
C···C	3.37	O···O	3.15
C···N	3.32	O···H	2.41
C···O	3.29	H···H	2.39
C···H	2.50		

ular contacts revealed by a distance scan * are given in Table 4. No short N···O, N···N, or N···H contacts were found. The hydrogen atom positions have been assumed with a C—H distance equal to 1.05 Å.

The space group requires a planar central bond arrangement. The twist angles of the phenyl and nitro groups and the angle between the central carbon bonds may be influenced by the packing. The central bond angles do not differ significantly from 120°, but the determined twist angles show that there is a deviation from trigonal symmetry. Deviations of atomic positions from different least-squares planes are listed in Table 5. According to these calculations there is a propeller-shaped twist, the phenyl group containing the two-fold axis is twisted 40° about the central carbon bond, and the phenyl groups in general positions have twist angles of 30°. The nitro group in the special position is twisted another 20° in the same direction as the ring, while the NO_2 groups in general positions lie in the ring planes. The 30° twist angle of the two phenyl groups is in good agreement with the values found in carbonium ion structures,[13–15] and the angle is in both cases determined by the *ortho* hydrogen contacts. The excessive twist of the one ring and the additional

Table 5. Deviation in Å of atoms from least squares planes through, a: the two-fold axis, C_1', C_4', and N_1', b: the central carbon atom, the phenyl group, and the nitrogen atom in special position, and c: the central carbon atom, the phenyl group and the nitrogen atom in general position.

a	Å	c	Å
C_1'	-0.01_9	C_0'	-0.00_5
C_4'	$+0.04_5$	C_1'	-0.01_2
N_1'	-0.02_8	C_2'	$+0.02_0$
		C_3'	-0.01_4
		C_4'	$+0.02_6$
b		C_5'	$+0.00_8$
	Å	C_6'	-0.00_4
C_2	$+0.02_8$	N_1'	-0.01_8
C_3	-0.02_8	O_1'	$+0.11_1$
O_1	-0.37_0	O_2'	$+0.02_8$

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* Program written at University of California, Los Angeles.

twist of the nitro group is caused by the packing. The ring and the nitro group were found by Trotter to be coplanar in nitrobenzene. The oxygen atoms of the nitro group in general position are both found close to the least-squares plane c in Table 5; they are, however, both on one side. The results of the analysis of the anisotropic thermal parameters (Table 6) may indicate a statistical distribution. The normal of the least-squares plane through the two-fold axis, C_1', C_4', and N′ makes an angle of 20° with the *a* axis and is less than 3° from the [101] direction.

Table 6. The principal axes of the atomic vibrations given by components of a unit vector in fractional coordinates e_x, e_y, e_z, the corresponding root mean square amplitude of vibration $(\overline{u^2})^{\frac{1}{2}}$ (Å) and B (Å²).

Atom	$(\overline{u^2})^{\frac{1}{2}}$	B	e_x	e_y	e_z
	0.36	10.31	0.092	−0.050	0.021
O_1	0.22	3.98	−0.029	0.029	0.049
	0.16	1.99	0.084	0.064	−0.006
	0.50	19.73	0.105	0.000	0.031
O_1'	0.32	7.86	0.031	0.079	−0.019
	0.20	3.22	−0.067	0.037	0.040
	0.35	9.84	0.127	0.007	0.007
O_2'	0.29	6.65	−0.016	0.082	0.016
	0.24	4.60	0.014	0.027	−0.051

In the gas electron-diffraction investigation of triphenylmethyl[1] the central CCC angles were determined to be 116—118°. However, it was pointed out that one could not distinguish between a real deviation from planarity and a planar model with a shrinkage effect[16] owing to the thermal vibrations. In this crystal structure no particularly short intermolecular distances have been found which could suggest that a slight deviation from planarity should have been present as found in triphenylmethyl. The planarity found in this investigation indicates that the deviation found in gas electron diffraction was apparent and due to thermal motion. A determination of the carbon-hydrogen distances would have been of great interest for comparison with the apparent lengthening of the C—H bond length found in triphenylmethyl. More and better data would be necessary to obtain information about the hydrogen positions in the present radical.

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