The Crystal Structure of the Phenyl iso*Cyanate Dimer.*

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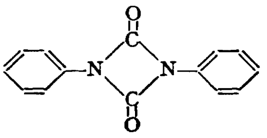
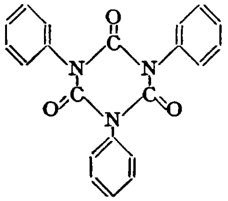
The crystal structure of the phenyl *iso*cyanate dimer has been determined from *X*-ray diffraction data. A complete three-dimensional analysis has been carried out, and from the resulting atomic co-ordinates, the geometry of the molecule has been thoroughly investigated. The dimer is found to be isostructural with *p*-terphenyl.

The dimer and the trimer formed by self-condensation of phenyl *iso*cyanate have long been known (Hofmann, *Annalen*, 1861, Suppl. **1**, 57), and although their formulæ have been assumed to be (I) and (II) respectively, confirmation by physical methods was desirable. Further, if (I) were correct it would be of interest to determine the bond lengths and inter-bond angles accurately as no similar structure has yet been worked out. The dimensions of the triazine ring in the trimer (II) would be of less interest as these data are already available from compounds such as cyanuric acid (Wiebenga, *J. Amer. Chem. Soc.*, 1952, **74**, 6156), cyanuric triazide (Knaggs, *Proc. Roy. Soc.*, 1935, *A*, **150**, 576), and melamine (Hughes, *J. Amer. Chem. Soc.*, 1941, **63**, 1737). Accordingly preference was given to the dimer.

(I)





(II)

The unit-cell dimensions of the dimer are $a = 12{\cdot}77$, $b = 5{\cdot}50$, $c = 8{\cdot}09$ Å, and the angle $\beta = 92°$. Assuming two molecules per unit cell, and a molecular weight of 238·1 gives 1·393 as calculated specific gravity; that observed by flotation in sodium iodide solution was 1·400. Absent reflexions were $\{0k0\}$ for k odd and $\{h0l\}$ for l odd, so the space group was fixed unambiguously as $P2_1/c$ (C_{2h}^5). This requires that the dimeric molecule shall be centro-symmetrical, and that the centres of the molecules should coincide with the centres of symmetry in the unit cell. The unit cell dimensions of the trimer are $a = 12{\cdot}67$, $b = 13{\cdot}76$, $c = 9{\cdot}83$ Å, and $\beta = 90°$. Absent reflexions indicate body-centering, with space group either Ia or I2/a. The specific gravity of the sample available was rather difficult to determine, the best value being only 1·34 compared with 1·38 required for four molecules per unit cell. Owing to the large number of atoms, the possible absence of a centre of symmetry, and the size of the unit cell which would preclude the resolution of individual atoms in projection, no further work was done on the trimer.

Determination of the Structure.—The unit-cell dimensions of the dimer bore a close resemblance to those of *p*-terphenyl (Pickett, *Proc. Roy. Soc.*, 1933, *A*, **142**, 333) and, as the space groups were the same, it was probable that the molecular packing in the two structures would be identical. The way in which organic compounds of similar molecular shape may crystallise in an isostructural series has been noted before (Brown, *Acta Cryst.*, 1951, **4**, 100) and Table 1 shows the crystal data for some of these substances.

In each of these crystals, the two unit-cell dimensions *b* and *c* correspond very closely; these are determined by the closest lateral packing of benzene rings. The other dimension *a* and the angle β depend on the length of the molecule and the nature and size of the *para*-substituents. Use was made of this analogy in the determination of the structure of the phenyl *iso*cyanate dimer. After due allowance for the small difference in length of the molecules, the co-ordinates worked out for *p*-terphenyl (Pickett, *loc. cit.*) were given to the dimer. There was at once moderately good agreement between the calculated and the experimental structure amplitudes for the *h0l* planes of the highest spacing, and two stages of Fourier projections were adequate to complete the refinement of this zone. In the same way, by using the *y* co-ordinates from the *p*-terphenyl structure, satisfactory agreement was obtained for F{*hkl*} of the dimer, and two stages of three-dimensional Fourier syntheses computed along appropriate lines and over appropriate sections, with finally all the 726 terms, were sufficient to complete the refinement of all the atomic co-ordinates. These are given in Table 2, and a composite electron-density map showing the whole molecule as a projection on the (011) plane is reproduced in Fig. 1.

FIG. 1. *Composite three-dimensional electron density map represented as a projection on the* (011) *plane. The contours are at intervals of* 1 e/Å^3, *commencing at* 2 e/Å^3. *The origin is at the centre of the molecule.*

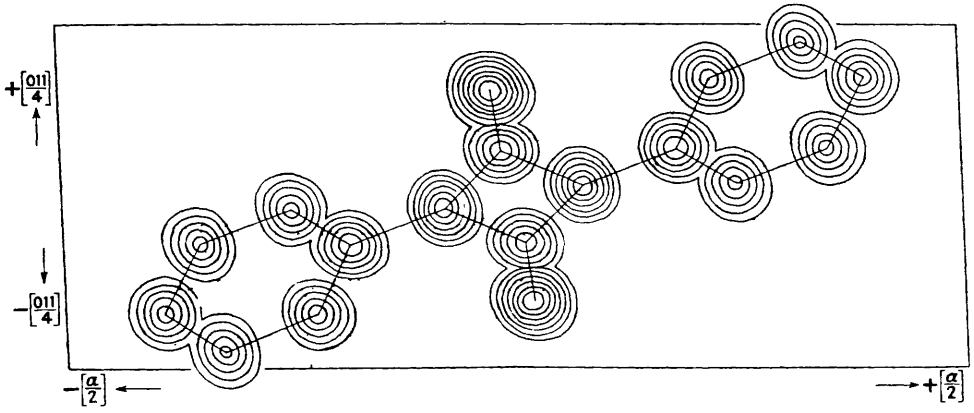


TABLE 1. *Cell dimensions of some analogous structures.*

	a	*b*	*c*	*β*
Phenyl *iso*cyanate dimer	12·77	5·50	8·09	92°
p-Terphenyl	13·59	5·60	8·08	92°
Diphenyl	9·57	5·67	8·11	94½°
4 : 4′-Diphenyldiphenyl	18·4	5·64	8·14	97°
γ-Quinol	13·24	5·20	8·11	107°

TABLE 2. *Atomic co-ordinates of the dimer.**

	x	*y*	*z*		*x*	*y*	*z*		*x*	*y*	*z*
C_1	0·184	$\bar{1}$·994	0·073	C_4	0·393	$\bar{1}$·997	0·173	C_7	0·012	0·160	$\bar{1}$·931
C_2	0·247	0·174	0·021	C_5	0·328	$\bar{1}$·822	0·228	N	0·078	$\bar{1}$·991	0·017
C_3	0·352	0·176	0·071	C_6	0·224	$\bar{1}$·818	0·173	O	0·023	0·330	$\bar{1}$·850

* For numbering see Fig. 2.

TABLE 3. *Experimental and calculated structure amplitudes.*

hkl	F(exp.)	F(calc.)	*hkl*	F(exp.)	F(calc.)	*hkl*	F(exp.)	F(calc.)	*hkl*	F(exp.)	F(calc.)
100	31	+47	806	7	+ 8	61$\bar{3}$	4	+ 5	10,2,0	8	+10
200	6	− 6	906	3	− 3	51$\bar{3}$	8	−12	11,2,0	7	+ 7
300	51	+50	10,0,$\bar{8}$	3	− 5	41$\bar{3}$	3	+ 4	13,2,0	5	+ 3
400	17	+16	80$\bar{8}$	2	− 4	31$\bar{3}$	3	+ 2	15,2,0	5	+ 4
500	40	+34	70$\bar{8}$	6	− 9	21$\bar{3}$	17	+20	13,2,$\bar{1}$	5	− 3
600	11	+10	60$\bar{8}$	5	− 4	11$\bar{3}$	52	+38	12,2,$\bar{1}$	6	− 5
700	17	−16	50$\bar{8}$	4	− 3	013	15	+11	11,2,$\bar{1}$	7	+ 8
800	6	+ 7	00$\bar{8}$	7	− 6	113	22	+15	92$\bar{1}$	12	−12
900	8	+11	508	12	−14	213	2	0	82$\bar{1}$	5	+ 6
10,0,0	4	+ 2	608	5	− 5	313	3	+ 3	72$\bar{1}$	8	+10
11,0,0	4	+ 3	708	5	+ 9	413	16	+16	62$\bar{1}$	2	0
12,0,0	7	+11	4,0,$\bar{10}$	3	+ 3	513	6	− 6	52$\bar{1}$	5	+ 5
13,0,0	4	+ 4	2,0,10	3	− 3	613	7	− 6	42$\bar{1}$	2	− 2
15,0,0	2	+ 2	3,0,10	2	− 3	713	6	− 5	32$\bar{1}$	11	+ 8
15,0,$\bar{2}$	3	− 3	4,0,10	4	− 6	813	4	− 2	22$\bar{1}$	22	+23
11,0,$\bar{2}$	7	+ 6	310	8	−12	913	4	+ 4	021	2	+ 6
10,0,$\bar{2}$	11	−13	410	8	−12	11,1,3	7	+ 8	121	13	−14
90$\bar{2}$	16	−14	510	10	−11	10,1,$\bar{4}$	4	− 2	221	3	− 5
80$\bar{2}$	14	−15	610	8	−11	51$\bar{4}$	3	− 5	321	5	+ 2
70$\bar{2}$	18	−18	810	10	−13	41$\bar{4}$	8	+12	421	2	− 3
60$\bar{2}$	5	0	910	13	−19	31$\bar{4}$	12	+16	10,2,1	3	+ 3
50$\bar{2}$	24	−19	12,1,0	7	−12	21$\bar{4}$	12	−19	13,2,$\bar{2}$	6	+ 6
40$\bar{2}$	18	−11	13,1,0	4	− 5	11$\bar{4}$	6	− 7	12,2,$\bar{2}$	5	+ 5
30$\bar{2}$	16	−16	13,1,$\bar{1}$	5	+ 6	014	20	−20	11,2,$\bar{2}$	7	+ 5
20$\bar{2}$	16	−13	12,1,$\bar{1}$	4	+ 5	114	5	+ 5	82$\bar{2}$	3	− 2
10$\bar{2}$	83	+74	11,1,$\bar{1}$	7	+ 9	214	9	+10	72$\bar{2}$	4	− 4
002	33	+23	10,1,$\bar{1}$	3	+ 3	314	7	+ 6	62$\bar{2}$	9	+ 9
102	20	−17	91$\bar{1}$	3	− 3	414	12	+11	52$\bar{2}$	5	− 6
202	28	+20	81$\bar{1}$	2	− 2	514	6	− 7	42$\bar{2}$	5	− 5
402	38	+25	71$\bar{1}$	8	−12	614	3	+ 3	32$\bar{2}$	10	+ 8
502	17	+13	61$\bar{1}$	3	+ 7	714	19	+22	22$\bar{2}$	7	+ 6
602	21	−17	51$\bar{1}$	3	+ 4	914	4	− 5	12$\bar{2}$	25	+19
702	6	+ 6	41$\bar{1}$	7	− 8	10,1,4	7	+10	022	17	+12
802	20	+18	31$\bar{1}$	5	+ 7	11,1,4	6	+ 6	122	22	+14
902	6	+ 6	21$\bar{1}$	11	−18	12,1,4	3	+ 3	222	9	− 6
10,0,2	7	+ 4	11$\bar{1}$	44	+46	13,1,4	2	+ 3	322	21	−20
11,0,2	16	+21	011	63	+61	81$\bar{5}$	4	− 7	422	2	+ 2
12,0,2	12	+10	111	16	−17	61$\bar{5}$	9	−16	522	11	−10
13,0,2	3	+ 3	211	7	+ 4	51$\bar{5}$	7	−10	622	6	− 6
14,0.2	7	+ 7	311	10	− 9	21$\bar{5}$	7	+ 6	722	14	−13
15,0,2	2	+ 3	411	2	− 4	11$\bar{5}$	7	+ 6	822	25	−26
12,0,$\bar{4}$	12	+15	511	5	+ 9	015	9	+ 9	922	5	+ 5
11,0,$\bar{4}$	4	− 7	611	18	−19	115	7	− 7	10,2,2	8	+ 7
10,0,$\bar{4}$	10	−13	711	11	−12	215	3	+ 3	11,2,2	5	− 5
80$\bar{4}$	6	− 8	811	5	− 5	315	6	+ 6	13,2,2	5	− 4
60$\bar{4}$	11	−10	911	3	− 2	615	9	− 9	15,2,2	3	+ 2
50$\bar{4}$	27	−24	10,1,1	3	+ 4	715	7	− 8	14,2,$\bar{3}$	3	− 4
20$\bar{4}$	4	− 1	11,1,1	3	+ 3	815	7	+ 8	13,2,$\bar{3}$	5	− 6
004	9	+ 3	12,1,1	7	+ 7	915	4	+ 3	11,2,$\bar{3}$	5	− 4
104	9	− 8	13,1,$\bar{2}$	5	− 6	10,1,5	5	+ 5	10,2,$\bar{3}$	12	−15
204	26	−23	11,1,$\bar{2}$	5	+ 9	11,1,5	3	+ 3	92$\bar{3}$	6	− 6
304	8	− 3	10,1,$\bar{2}$	7	−13	13,1,5	4	+ 5	72$\bar{3}$	8	− 9
404	14	− 9	91$\bar{2}$	7	−13	416	4	− 5	62$\bar{3}$	7	− 8
504	19	−17	81$\bar{2}$	5	+ 7	516	4	− 4	52$\bar{3}$	13	−15
604	13	−12	71$\bar{2}$	3	− 6	616	6	+ 6	42$\bar{3}$	9	−10
704	15	−15	61$\bar{2}$	7	−11	816	6	− 8	32$\bar{3}$	14	+14
804	4	− 3	51$\bar{2}$	9	−15	017	5	− 5	22$\bar{3}$	10	−12
10,0,4	3	+ 3	41$\bar{2}$	20	−30	117	7	− 7	12$\bar{3}$	28	−28
11,0,4	4	+ 5	31$\bar{2}$	5	+ 5	317	5	− 6	023	12	−11
13,0,4	2	+ 2	11$\bar{2}$	76	−61	417	3	− 2	123	8	− 6
13,0,$\bar{6}$	5	+ 9	012	36	+24	517	7	−10	223	16	+12
11,0,$\bar{6}$	6	− 8	112	6	− 5	617	10	−14	423	4	+ 4
90$\bar{6}$	3	− 2	212	40	+31	020	5	− 5	523	5	− 4
60$\bar{6}$	10	−14	312	22	+23	220	12	+ 9	623	10	−12
40$\bar{6}$	8	+12	412	20	+17	320	21	−23	723	7	+ 7
20$\bar{6}$	3	+ 2	512	17	+17	420	22	−25	923	11	−11
10$\bar{6}$	18	+19	712	7	+ 8	520	5	− 1	92$\bar{4}$	7	− 7
006	7	+ 7	812	9	+12	620	13	−14	82$\bar{4}$	7	− 7
106	12	−10	12,1,$\bar{3}$	4	+ 8	720	6	− 4	72$\bar{4}$	3	+ 5
606	23	−24	10,1,$\bar{3}$	6	−10	820	11	−12	62$\bar{4}$	3	− 4
706	4	− 4	81$\bar{3}$	5	− 6	920	15	−18	42$\bar{4}$	4	− 2

TABLE 3. (*Continued.*)

hkl	F(exp.)	F (calc.)	*hkl*	F(exp.)	F(calc.)	*hkl*	F(exp.)	F(calc.)	*hkl*	F(exp.)	F(calc.)
$32\bar{4}$	7	−6	128	3	+2	433	7	−8	$74\bar{1}$	5	−7
$22\bar{4}$	29	+28	228	5	+5	533	12	−11	$64\bar{1}$	2	+1
$12\bar{4}$	24	+18	328	3	−5	633	4	−3	$54\bar{1}$	2	−3
024	7	+3	428	4	−4	733	18	−19	$44\bar{1}$	2	+2
124	11	+9	528	3	−4	833	11	−12	$34\bar{1}$	2	−2
224	5	−4	628	3	−2	933	11	+10	$24\bar{1}$	5	−12
324	18	+10	728	3	−3	$43\bar{4}$	3	−2	141	6	+8
424	8	+5	928	3	+3	$13\bar{4}$	5	−1	241	4	+5
524	5	−4	$42\bar{9}$	5	+7	034	12	+5	10,4,1	4	−4
624	5	−3	$32\bar{9}$	2	+2	134	8	+2	13,4,1	3	−2
724	12	−11	$12\bar{9}$	4	+4	234	3	0	$11,4,\bar{2}$	3	+5
824	4	+4	029	4	−3	334	6	+1	$84\bar{2}$	2	−2
924	8	+7	129	4	−4	434	5	+1	$74\bar{2}$	2	−2
11,2,4	5	+4	229	2	−4	534	6	+3	$64\bar{2}$	2	+5
$82\bar{5}$	4	−2	429	2	−2	634	5	+1	$54\bar{2}$	2	−4
$72\bar{5}$	5	−4	529	5	−6	734	5	−3	$44\bar{2}$	5	−4
$62\bar{5}$	5	−7	230	3	+1	$12,3,\bar{5}$	4	+4	$14\bar{2}$	8	+12
$52\bar{5}$	7	−7	330	5	+3	$11,3,\bar{5}$	6	+7	242	3	−2
$42\bar{5}$	3	−1	$11,3,\bar{1}$	5	+5	$10,3,\bar{5}$	4	+4	342	7	−9
$32\bar{5}$	6	−7	$10,3,\bar{1}$	3	−3	$93\bar{5}$	3	−3	542	4	−6
$22\bar{5}$	17	−17	$93\bar{1}$	15	−17	$83\bar{5}$	5	+5	642	4	−7
$12\bar{5}$	11	−7	$83\bar{1}$	6	−6	$73\bar{5}$	5	+6	742	6	−6
025	10	+5	$73\bar{1}$	7	−8	$63\bar{5}$	3	+3	842	7	−12
125	19	+15	$63\bar{1}$	5	−5	$53\bar{5}$	6	+8	$10,4,\bar{3}$	5	+9
225	15	+11	$53\bar{1}$	4	−4	$43\bar{5}$	9	−9	$74\bar{3}$	2	+6
325	18	+13	$43\bar{1}$	20	−22	$33\bar{5}$	9	+8	$64\bar{3}$	2	+4
425	13	+9	$23\bar{1}$	12	+14	$23\bar{5}$	28	+29	$54\bar{3}$	7	+10
625	14	+12	$13\bar{1}$	4	+1	135	3	+4	$44\bar{3}$	5	+7
725	12	+12	031	3	−1	335	7	+7	$34\bar{3}$	5	−10
825	5	−4	131	15	+20	435	8	−6	$24\bar{3}$	7	+9
10,2,5	7	+7	231	10	+10	535	10	−10	$14\bar{3}$	12	+16
$92\bar{6}$	7	−6	331	14	−18	635	8	−7	043	8	+9
$62\bar{6}$	3	−2	531	2	+1	735	8	−7	143	8	+7
$52\bar{6}$	6	−5	731	4	+6	12,3,5	4	−4	243	2	−5
$42\bar{6}$	12	−13	831	20	−21	$43\bar{6}$	5	+1	443	2	−2
$32\bar{6}$	14	+14	931	3	−4	$33\bar{6}$	7	+1	543	6	+5
$22\bar{6}$	14	+13	10,3,1	16	+13	336	5	+1	643	7	+8
$12\bar{6}$	7	−7	$14,3,\bar{2}$	2	0	436	6	+2	743	3	−5
126	6	−3	$12,3,\bar{2}$	3	−1	$83\bar{7}$	5	+3	843	3	−3
226	3	+3	$11,3,\bar{2}$	3	−3	$53\bar{7}$	4	−4	943	4	+7
326	5	+5	$93\bar{2}$	3	+1	$43\bar{7}$	5	+3	$74\bar{4}$	6	+4
426	10	−9	$83\bar{2}$	5	−3	$33\bar{7}$	20	+21	$24\bar{4}$	12	+14
526	9	−6	$73\bar{2}$	2	0	$23\bar{7}$	9	+6	$14\bar{4}$	7	+8
626	5	−3	$63\bar{2}$	2	0	$13\bar{7}$	5	+3	144	4	+4
926	3	+3	$43\bar{2}$	3	+3	037	9	+9	344	3	+4
10,2,6	6	+5	$13\bar{2}$	7	+5	137	6	+5	544	3	−3
$72\bar{7}$	3	+4	132	9	+5	237	16	+13	744	7	−7
$62\bar{7}$	5	+4	232	5	+1	337	6	+5	$54\bar{5}$	4	+4
$52\bar{7}$	3	0	832	7	−3	437	5	−4	$34\bar{5}$	6	+6
$42\bar{7}$	4	+3	932	7	−2	637	4	+5	$24\bar{5}$	9	+11
$32\bar{7}$	8	+8	$14,3,\bar{3}$	5	+5	937	6	+6	145	7	−8
227	4	+1	$13,3,\bar{3}$	7	+6	040	5	+5	245	6	−6
327	6	+2	$93\bar{3}$	5	−5	240	2	−2	345	6	−7
427	5	+2	$83\bar{3}$	9	−11	340	6	−9	645	8	−8
527	4	+3	$73\bar{3}$	5	−3	440	6	−12	745	8	−9
627	5	+4	$63\bar{3}$	5	−4	640	5	−8	10,4,5	5	−5
727	2	+1	$53\bar{3}$	13	−13	740	6	−6	$34\bar{6}$	7	+7
827	2	+2	$43\bar{3}$	9	−7	840	7	−7	$24\bar{6}$	7	+7
927	3	+3	$33\bar{3}$	12	−13	940	5	−8	046	4	+3
10,2,7	2	+3	$13\bar{3}$	7	+7	$13,4,\bar{1}$	3	+3	246	5	+1
$52\bar{8}$	4	−4	033	8	−7	$11,4,\bar{1}$	7	−7	346	5	+3
$42\bar{8}$	6	+6	133	4	−3	$10,4,\bar{1}$	2	−1			
$32\bar{8}$	9	+9	233	21	−16	$94\bar{1}$	4	+7			
$12\bar{8}$	3	+3	333	13	−12	$84\bar{1}$	4	−5			

Description of the Structure.—The crystal structure of the phenyl *iso*cyanate dimer comprises discrete molecules, each of which has a centre of symmetry relating the two halves of the molecule. There is no intermolecular bonding other than van der Waals contacts, of which the closest are O–O′ = 3·23, C_3–C_5' = 3·90, C_2–C_6' = 3·91, C_3–C_4' = 3·98 and C_4–$C_4' = 3{\cdot}98$ Å. The interatomic bond lengths and inter-bond angles calculated from the co-ordinates listed in Table 2 are shown diagrammatically in Fig. 2. By Cruickshank's method (*Acta Cryst.*, 1949, **2**, 65), the mean standard deviation of the position co-ordinates $\sigma = 0{\cdot}010$ Å for the carbon atoms, hence the standard deviation of a bond is 0·014 Å, and the Gaussian probable error is ±0·010 Å for the bond lengths and ±1° for the inter-bond angles. The mean C–C bond length in the benzene ring (1·37 Å) is a little, but not significantly, shorter than the usual value (1·385 Å), that for C_1–N agrees with that found in acetanilide (1·426 Å) (Brown and Corbridge, *ibid.*, 1954. 7, 711), and that for C_7–O is the same as in ethylene carbonate (Brown, *loc. cit.*, p. 92). The two pairs of C_7–N bonds in the central four-membered ring, which might be expected to be equivalent, are different by 0·07 Å, for which no satisfactory explanation can be given. The angles associated with the benzene ring have 120° as their mean value. The other angles approximate to 90° within the four-membered ring, and 135° externally to it.

The molecule as a whole is not planar. The benzene rings are individually planar, the equation of the mean plane through one of them, derived by least squares, and referred to rectangular axes, being

$$0{\cdot}252x' - 0{\cdot}553y - 0{\cdot}794z' = 0{\cdot}135$$

from which the atoms are displaced by the following amounts: C_1 0; C_2 −0·006; C_3 +0·002; C_4 +0·016; C_5 −0·021; C_6 +0·014 Å. The central four-membered ring is necessarily planar, but the C=O bond makes an angle of 87·5° with its normal. The equation of the mean plane through the origin, C_7, N, and O is

$$0{\cdot}088x' - 0{\cdot}555y - 0{\cdot}827z' = 0.$$

The normals to these two planes intersect at an angle of 9·5°. The C_1–N bond makes an angle of 79° with the normal to the central ring, the nitrogen atom being slightly pyramidal. This distortion from planarity may be due to the close approach of the oxygen atom to the benzene ring. If the whole system were rigidly planar, the distances between O and C_2, and between O′ and C_6 would be 3·22 and 3·24 Å respectively, but the distortions increase these distances to 3·25 and 3·26 Å. The differences are very slight, but since the true contacts are between the oxygen atoms and the hydrogen atoms attached to the carbon atoms, it may be that in this way adequate clearance is given to the hydrogen atoms.

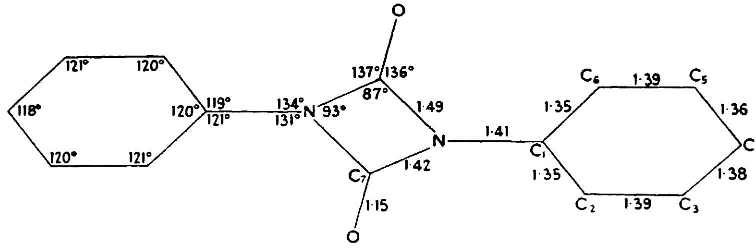


FIG. 2. *Diagrammatic representation of the molecule showing bond lengths and inter-bond angles.*

EXPERIMENTAL

Crystals of the dimer were prepared by Mr. A. C. Farthing of these laboratories by treating phenyl *iso*cyanate with pyridine, and recrystallising the product from benzene. The crystals were mostly platy with (100) tabular, and m. p. 176·2°, although some were acicular with [011] as the needle axis. No good cleavage was observed, but sections of larger crystals were cut to suitable dimensions for *X*-ray photography. The unit-cell dimensions were obtained by measurement of layer lines on rotation photographs. *X*-Ray intensity data were obtained from Weissenberg moving-film photographs about [b] (zero and four layers), and [c] (zero layer only). Multiple films were used for increasing the intensity range, and the densities of the spots were estimated visually by comparison with a time-exposure calibrated film strip. Owing to the platy shape of the crystals, the accuracy of the intensities was not thought to be very high. After correction for the usual polarisation and geometrical factors, the structure amplitudes were converted to the absolute scale by comparison with calculated values.

The atomic scattering factors used finally were derived experimentally, but corresponded approximately to the values of International Tables, with temperature factor B = 3·5 Å^2.

The precise values were :

$\sin\theta/\lambda$	0	0·1	0·2	0·3	0·4	0·5	0·6	0·7
$4f_c$	24·0	17·0	10·8	7·0	4·6	2·9	1·8	1·3

The residual $R = \dfrac{\sum|F(\text{exp.}) - F(\text{calc.})|}{\sum|F(\text{exp.})|}$ summed over all values of F{*hkl*} except those where F(exp.) = 0 was 0·21. Values of calculated and experimental structure amplitudes are given in Table 3; where F(exp.) = 0, these are omitted, but in these instances the calculated structure amplitudes were all small. No attempt has been made to locate the hydrogen atoms, nor were they allowed for in the structure-amplitude calculations. Neither has any correction been made for errors due to the artificial termination of the Fourier series, but in view of the appreciable temperature factor it is not expected that inaccuracies due to this cause will be very great.

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