References

Barrett, R. M. & Steele, D. (1972). *J. Mol. Struct.* 11, 105–108.

Bastiansen, O. & Trætteberg, M. (1962). *Tetrahedron*, 17, 147–154.

Baudour, J. L. (1972). *Acta Cryst.* B28, 1649–1656.

Baudour, J. L., Delugeard, Y. & Cailleau, H. (1976). *Acta Cryst.* B32, 150–154.

Bree, A., Pang, C. Y. & Rabeneck, I. (1971). *Spectrochim. Acta*, 27a, 1293–1297.

Cailleau, H. (1976). Private communication.

Charbonneau, G. P. & Delugeard, Y. (1976). *Acta Cryst.* B32, 1420–1423.

Friedman, P. S., Kopelman, R. & Prasad, P. N. (1974). *Chem. Phys. Lett.* 24, 15–17.

Hargreaves, A. & Rizvi, S. H. (1962). *Acta Cryst.* 15, 365–373.

Kay, M. J. & Behrendt, D. R. (1963). *Acta Cryst.* 16, 157–162.

Robertson, G. B. (1961). *Nature, Lond.* 191, 593–594.

Schmid, E. D. & Brosa, B. (1972). *J. Chem. Phys.* 56, 6267–6268.

Trotter, J. (1961). *Acta Cryst.* 14, 1135–1140.

N-(2-Imidazol-4-ylethyl)phthalimide

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Abstract. Monoclinic, $P2_1/a$, $a = 18\cdot434\,(9)$, $b = 7\cdot343\,(2)$, $c = 18\cdot002\,(7)$ Å, $\beta = 91\cdot38\,(4)°$, $C_{13}H_{11}N_3O_2$, $Z = 8$, $D_x = 1\cdot315$ g cm^{-3}. The molecules are arranged in infinite NH $\cdots$ N hydrogen-bonded chains with N $\cdots$ N distances of $2\cdot79$ and $2\cdot84$ Å between imidazolyl groups belonging to symmetry-independent molecules. Parallel stacking of phthalimide groups as well as of alternating phthalimide and imidazolyl groups occurs in the structure.

Introduction. Crystals of $C_{13}H_{11}N_3O_2$ were grown as colorless, fragile plates from an ethyl acetate–petroleum spirit solution. A crystal, approximately $0\cdot2 \times 0\cdot2 \times 0\cdot05$ mm, was used in the X-ray work. Cell parameters and intensity data were measured with a Syntex $P\bar{1}$ diffractometer with graphite-monochromatized Cu $K\alpha$ radiation. 3350 reflections with $\theta < 70°$ were measured. The diffractometer was operated in the θ–2θ variable-speed scan mode, in which the speed of the scan is dependent on a peak-height preview of the reflection intensity. High-intensity reflections were collected at 24° min^{-1} (2θ), low-intensity reflections at $0\cdot5°$ min^{-1} and medium-intensity reflections at intermediate scan speeds. Scan ranges (2θ) extended from $\alpha_1 - 1°$ to $\alpha_2 + 1°$; stationary background counts were made at the ends of the scans. 1392 reflections with $I > 3\sigma(I)$ were employed in the structure solution and subsequent refinement. Systematic absences of $h0l$ for

h odd and of $0k0$ for *k* odd uniquely identified the space group as $P2_1/a$.

The structure was solved by direct methods with the program *PHASE* (Stewart, 1970), and refined by means of the least-squares program *CLS* (Schilling, 1970). The scattering factors used were those given in *International Tables for X-ray Crystallography* (1968). The weights were taken as $w = [\sigma(F_o) + \alpha|F_o|^2]^{-2}$ where $\sigma(F_o)$ was determined from counting statistics, and α is an empirically adjusted parameter with the optimal value of $6\cdot0 \times 10^{-5}$. A secondary extinction parameter, β, was refined and applied to the final structure factor magnitudes by means of the relation (Zachariasen, 1968):

$$|F_c^{corr}| = |F_c|[1 + \beta|F_c|^2 p_2/(p_1 \sin 2\theta)]^{-0\cdot25}$$

where $p_n = (1 + \cos^{2n} 2\theta)/2$. The value of β was determined to be $0\cdot377 \times 10^{-5}$. The final R value was $0\cdot046$ for the 1392 reflections used in the refinement. The positional parameters of the C, N and O atoms are given in Table 1† and the parameters of the H atoms in Table 2.

Discussion. From kinetic studies of the effect of imidazole on the rate of hydrolysis of *N*-methylphthalimide, Su & Shafer (1969a) inferred the existence

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of an addition compound in which a carbonyl C of the *N*-methylphthalimide moiety tends toward tetrahedral configuration through interaction with an N atom of imidazole. A similar proposal of an imidazole–phthalimide complex in aqueous solution had previously been made by Champy-Hatem (1966) on the basis of spectral studies. In a subsequent study of the hydrolysis of *N*-(2-imidazol-4-ylethyl)phthalimide, Su & Shafer (1969*b*) found evidence for a similar intramolecular interaction between the imidazolyl group and a phthalimide carbonyl, suggesting that a substantial fraction of the compound exists in a cyclomeric form in aqueous solution. The crystallographic work was inspired by the thought that such a form might exist in the solid as well.

The cyclic form was not found in the crystalline state, however, as both independent molecules, *A* and *B*, are in the open, extended form. Conformationally,

the two molecules are approximate mirror images of each other. The main difference between them is that the phthalimido group of molecule *A* is planar, while the phthalimido group of molecule *B* has a puckered

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *of the non-hydrogen atoms*

The estimated standard deviation, given in parentheses, refers to the last significant digit.

	x	*y*	*z*
C(1*A*)	−221 (2)	1669 (7)	2769 (2)
C(2*A*)	−451 (3)	1137 (9)	2065 (3)
C(3*A*)	−1185 (4)	834 (9)	1956 (3)
C(4*A*)	−1661 (3)	1038 (9)	2522 (3)
C(5*A*)	−1432 (3)	1604 (9)	3235 (3)
C(6*A*)	−704 (2)	1890 (7)	3333 (3)
C(7*A*)	−286 (2)	2469 (7)	4008 (2)
C(8*A*)	519 (3)	2092 (7)	3056 (3)
C(9*A*)	1030 (2)	3070 (7)	4312 (2)
C(10*A*)	1326 (3)	1419 (8)	4736 (3)
C(11*A*)	2601 (2)	1572 (8)	5337 (3)
C(12*A*)	2320 (2)	3081 (8)	6332 (3)
C(13*A*)	1884 (2)	1959 (7)	5313 (3)
N(1*A*)	437 (2)	2551 (6)	3797 (2)
N(2*A*)	1708 (2)	2911 (6)	5935 (2)
N(3*A*)	2877 (2)	2298 (6)	5992 (2)
O(1*A*)	−500 (2)	2817 (5)	4617 (2)
O(2*A*)	1090 (2)	2059 (5)	2741 (2)
C(1*B*)	2044 (3)	2750 (8)	−541 (3)
C(2*B*)	1307 (4)	2827 (9)	−405 (3)
C(3*B*)	1113 (4)	2603 (9)	329 (4)
C(4*B*)	1623 (4)	2337 (8)	877 (3)
C(5*B*)	2365 (4)	2269 (9)	749 (3)
C(6*B*)	2563 (3)	2483 (7)	8 (3)
C(7*B*)	3298 (3)	2558 (8)	−321 (3)
C(8*B*)	2424 (3)	2997 (8)	−1256 (3)
C(9*B*)	3739 (3)	3067 (8)	−1618 (3)
C(10*B*)	3999 (3)	1250 (8)	−1936 (3)
C(11*B*)	5231 (3)	1348 (8)	−2585 (3)
C(12*B*)	4816 (2)	2318 (8)	−3658 (2)
C(13*B*)	4499 (2)	1570 (7)	−2567 (2)
N(1*B*)	3167 (2)	2795 (6)	−1080 (2)
N(2*B*)	4241 (2)	2184 (6)	−3241 (2)
N(3*B*)	5422 (2)	1835 (6)	−3286 (2)
O(1*B*)	3887 (2)	2421 (7)	−29 (2)
O(2*B*)	2178 (2)	3302 (7)	−1857 (2)

Table 2. *Fractional coordinates* $(\times 10^3)$ *and isotropic thermal parameters of the hydrogen atoms*

The numbering follows that of the atoms to which the hydrogens are attached.

|	*x*	*y*	*z*	*B* (Å²)
H(2*A*)	−6 (3)	88 (8)	169 (3)	6·1 (15)
H(3*A*)	−141 (3)	51 (8)	144 (3)	8·4 (18)
H(4*A*)	−215 (3)	95 (8)	245 (3)	7·9 (17)
H(5*A*)	−172 (3)	175 (7)	366 (3)	6·1 (15)
H(9*A*)	142 (2)	359 (6)	399 (2)	4·1 (13)
H'(9*A*)	81 (2)	405 (7)	466 (2)	4·0 (12)
H(10*A*)	149 (2)	53 (7)	441 (3)	4·9 (14)
H'(10*A*)	91 (2)	86 (7)	500 (2)	4·9 (13)
H(11*A*)	289 (2)	99 (7)	498 (2)	4·6 (14)
H(12*A*)	233 (2)	377 (7)	685 (2)	4·9 (14)
H(N3*A*)	337 (2)	231 (7)	624 (3)	6·1 (15)
H(2*B*)	89 (3)	287 (9)	−90 (3)	8·6 (18)
H(3*B*)	51 (3)	262 (9)	40 (3)	10·0 (19)
H(4*B*)	144 (3)	216 (9)	138 (3)	8·9 (18)
H(5*B*)	282 (3)	192 (8)	109 (3)	7·9 (17)
H(9*B*)	421 (3)	359 (8)	−132 (3)	7·0 (16)
H'(9*B*)	354 (3)	390 (7)	−202 (3)	6·1 (15)
H(10*B*)	363 (3)	60 (7)	−211 (3)	5·4 (14)
H'(10*B*)	426 (3)	54 (8)	−151 (3)	6·5 (16)
H(11*B*)	557 (2)	102 (7)	−219 (2)	4·6 (14)
H(12*B*)	477 (2)	270 (7)	−421 (2)	4·5 (12)
H(N3*B*)	588 (2)	188 (6)	−350 (2)	2·6 (11)
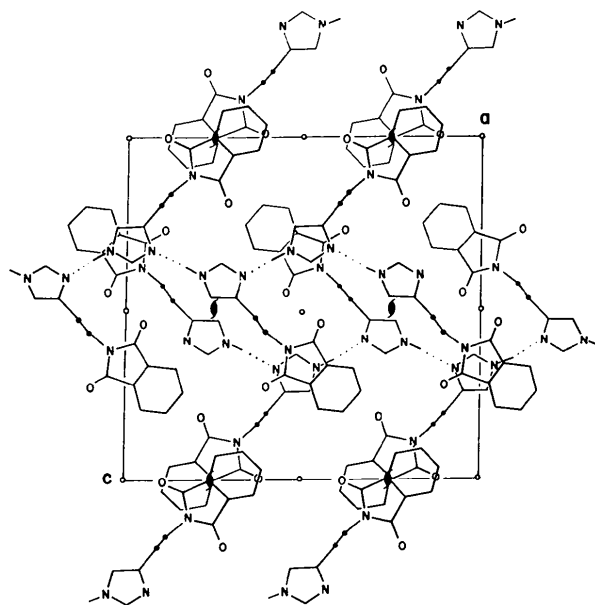
Fig. 1. Molecular packing in the title structure. The phthalimido group of molecule *B* lies on a 2_1 axis and overlaps with a symmetry-related phthalimido group, while the phthalimido group of molecule *A* overlaps with the imidazolyl group of molecule *B*. Hydrogen bonds are shown as dotted lines.
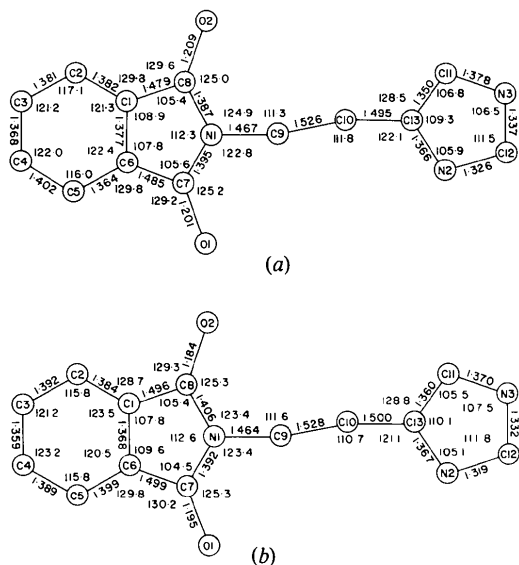
Fig. 2. Non-hydrogen bond lengths (Å) and angles (°) in (a) molecule A and (b) molecule B. Standard deviations are 0·006–0·008 Å for bond lengths, 0·4–0·6° for angles.

five-membered ring. This difference may be attributed to the different packing environments of the two phthalimido groups, as shown in Fig. 1. The phthalimido groups *A* stack in a closely spaced sandwich fashion with interleaving imidazolyl groups *B*. The phthalimido groups *B* stack in a looser, tilted manner with symmetry-related phthalimido groups *B*.

The imidazolyl groups of both molecules are planar and, as shown in Fig. 2, have bond lengths intermediate between single and double-bond values. This suggests that the imidazolyl groups consist of resonance hybrids of structures analogous to those invoked for imidazole itself (Martínez-Carrera, 1966).

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

	N···N	N–H	H···N	H–N···N
N(3A)–H···N(2B)	2·84	0·99	1·85	3
N(3B)–H···N(2A)	2·79	0·93	1·87	6
E.s.d.	0·01	0·04	0·04	4

Unusually short NH···N hydrogen bonds between imidazolyl N atoms join the molecules into infinite chains. Hydrogen-bond lengths and angles are given in Table 3. As in the imidazole structure the shortness of the NH···N hydrogen bonds may be attributed to dipole–dipole interactions between formally charged imidazolyl resonance species.

References

CHAMPY-HATEM, S. (1966). *Bull. Acad. Natl. Med.* (*Paris*), **150**, 137–142.

International Tables for X-ray Crystallography (1968). Vol. III. Birmingham: Kynoch Press.

MARTÍNEZ-CARRERA, S. (1966). *Acta Cryst.* **20**, 783–789.

SCHILLING, J. W. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 201–204. Copenhagen: Munksgaard.

STEWART, J. M. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 71–74. Copenhagen: Munksgaard.

SU, S. C. K. & SHAFER, J. A. (1969a). *J. Org. Chem.* **34**, 926–930.

SU, S. C. K. & SHAFER, J. A. (1969b). *J. Org. Chem.* **34**, 2911–2915.

ZACHARIASEN, W. H. (1968). *Acta Cryst.* A**24**, 212–216.