# Isatogens: Crystal Structure, Electron Density Calculations and $^{13}C$ Nuclear Magnetic Resonance Spectra.

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Material for deposition and scrutiny

1. List of atomic co-ordinates for 2-phenylisatogen (1a). for H atoms
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8. ~~Calculated structure factors for 2-(2-pyridyl)isatogen - computer print out.~~

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1. ATOMIC COORDINATES FOR 2-PHENYL ISATOGEN

(esd in parentheses)

| ATOM | x/a | y/b | z/c |
|---|---|---|---|
| N1 | 0.18180 (18) | 0.05864 (26) | 0.06569 (9) |
| O1 | 0.26879 (15) | -0.03808 (25) | 0.07170 (8) |
| O3 | -0.05301 (15) | 0.31493 (24) | 0.09801 (7) |
| C2 | 0.11020 (19) | 0.12620 (30) | 0.10585 (10) |
| C3 | 0.02593 (19) | 0.22884 (30) | 0.07743 (10) |
| C4 | 0.04894 (21) | 0.21797 (30) | 0.01685 (10) |
| C5 | 0.14435 (21) | 0.11478 (31) | 0.01041 (10) |
| C6 | 0.18988 (25) | 0.07427 (36) | -0.04029 (12) |
| C7 | 0.13452 (28) | 0.14781 (46) | -0.08648 (12) |
| C8 | 0.03753 (28) | 0.25097 (42) | -0.08040 (12) |
| C9 | -0.00675 (24) | 0.28842 (35) | -0.02811 (12) |
| C10 | 0.12040 (21) | 0.09632 (29) | 0.16559 (10) |
| C11 | 0.21581 (24) | 0.01176 (38) | 0.18862 (12) |
| C12 | 0.22244 (29) | -0.01368 (46) | 0.24573 (14) |
| C13 | 0.13502 (30) | 0.04399 (42) | 0.28054 (12) |
| C14 | 0.04188 (26) | 0.12692 (52) | 0.25806 (12) |
| C15 | 0.03244 (24) | 0.15427 (40) | 0.20150 (12) |
| H6 | 0.2662 | 0.0441 | -0.0445 |
| H7 | 0.1656 | 0.1073 | -0.1256 |
| H8 | 0.0039 | 0.2810 | -0.1207 |
| H9 | -0.0791 | 0.3804 | -0.0244 |
| H11 | 0.2764 | -0.0379 | 0.1630 |
| H12 | 0.2764 | -0.0788 | 0.2619 |
| H13 | 0.1394 | 0.0196 | 0.3232 |
| H14 | -0.0324 | 0.1530 | 0.2825 |
| H15 | -0.0440 | 0.2147 | 0.1828 |

2. ATOMIC COORDINATES FOR 2(2')-PYRIDYL ISATOGEN

(esd in parentheses)

| ATOM | x/a | y/b | z/c |
|---|---|---|---|
| N1 | 0.19200 (24) | 0.08267 (43) | 0.42522 (13) |
| O1 | 0.28486 (19) | 0.00004 (38) | 0.41754 (11) |
| O3 | -0.05228 (20) | 0.30319 (39) | 0.39279 (10) |
| C2 | 0.11891 (28) | 0.13926 (53) | 0.38476 (15) |
| C3 | 0.02802 (27) | 0.22794 (50) | 0.41495 (15) |
| C4 | 0.05162 (29) | 0.21436 (50) | 0.47681 (14) |
| C5 | 0.15207 (28) | 0.12284 (51) | 0.48324 (14) |
| C6 | 0.19877 (30) | 0.08304 (59) | 0.53479 (16) |
| C7 | 0.13950 (35) | 0.13742 (63) | 0.58319 (17) |
| C8 | 0.03885 (38) | 0.23120 (58) | 0.57840 (16) |
| C9 | -0.00553 (29) | 0.27010 (60) | 0.52478 (18) |
| C10 | 0.13437 (29) | 0.11903 (52) | 0.32360 (15) |
| C11 | 0.23764 (33) | 0.14127 (60) | 0.29702 (16) |
| C12 | 0.24694 (37) | 0.11106 (63) | 0.23928 (16) |
| C13 | 0.15461 (39) | 0.05176 (59) | 0.20893 (17) |
| C14 | 0.05575 (36) | 0.02758 (63) | 0.23828 (18) |
| N15 | 0.04282 (26) | 0.05510 (48) | 0.29485 (13) |
| H6 | 0.2837 | 0.0087 | 0.5389 |
| H7 | 0.1823 | 0.1927 | 0.6232 |
| H8 | 0.0265 | 0.2823 | 0.6206 |
| H9 | -0.0760 | 0.3380 | 0.5228 |
| H11 | 0.3085 | 0.1733 | 0.3249 |
| H12 | 0.3260 | 0.1395 | 0.2186 |
| H13 | 0.1611 | 0.0094 | 0.1607 |
| H14 | -0.0196 | -0.0324 | 0.2197 |

3. Equations of least-square mean planes for (1a) referred to orthogonal axes with displacements (A) of relevant atoms from planes.

(a) All non-H atoms

$0.5765\ X + 0.8135\ Y + 0.0768\ Z - 1.7132 = 0$

DISPLACEMENTS IN Å

| | | | |
|---|---|---|---|
| N1 | -0.0172 | C10 | 0.0009 |
| C2 | 0.0139 | C11 | 0.1352 |
| C3 | 0.0584 | O1 | -0.0473 |
| C4 | 0.0297 | O3 | 0.1230 |
| C5 | -0.0089 | C12 | 0.1221 |
| C6 | -0.0593 | C13 | -0.0243 |
| C7 | -0.0411 | C14 | -0.1530 |
| C8 | -0.0144 | C15 | -0.1452 |
| C9 | 0.0277 | | |

(b) PHENYL RING

$0.4782\ X + 0.8725\ Y + 0.1004 - 1.7173 = 0$

DISPLACEMENTS IN Å

| | | | |
|---|---|---|---|
| C10 | -0.0004 | C13 | -0.0010 |
| C11 | 0.0003 | C14 | 0.0009 |
| C12 | 0.0004 | C15 | -0.0002 |

(c) HETEROCYCLIC RING

$0.6003\ X + 0.7974\ Y + 0.0619\ Z - 1.7155 = 0$

| | | | |
|---|---|---|---|
| N1 | -0.0007 | C6 | -0.0048 |
| C2 | -0.0120 | C7 | 0.0055 |
| C3 | 0.0068 | C8 | -0.0095 |
| C4 | 0.0074 | C9 | -0.0027 |
| C5 | 0.0100 | | |

Dihedral angle between (b) & (c) 7.0(2.0)°

4. Equations of least squares mean planes for (1b) referred to orthogonal axes with displacements (Å) of relevant atoms from the planes.

(a) All non-H atoms

$0.3691X + 0.9268Y - 0.0700Z - 0.8083 = 0$

Atomic Displacements

| | | | |
|---|---|---|---|
| N1 | -0.0843 | C9 | 0.1793 |
| C2 | 0.0506 | C10 | 0.0225 |
| C3 | 0.2137 | C11 | 0.7298 |
| C4 | 0.1232 | C12 | 0.6561 |
| C5 | -0.0771 | C13 | -0.1103 |
| C6 | -0.2302 | C14 | -0.7602 |
| C7 | -0.1944 | N15 | -0.7191 |
| C8 | 0.0179 | O3 | 0.4159 |
| O1 | -0.2336 | | |

(b) PYRIDYL RING

$-0.2578X + 0.9493Y - 0.1800Z + 0.9779 = 0$

Atomic Displacements

| | | | |
|---|---|---|---|
| C10 | -0.0053 | C13 | -0.0060 |
| C11 | 0.0030 | C14 | 0.0041 |
| C12 | 0.0025 | N15 | 0.0017 |

(c) HETEROCYCLIC RING

$0.4955X + 0.8683Y - 0.0224Z - 1.4299 = 0$

Atomic Displacements

| | | | |
|---|---|---|---|
| N1 | 0.0180 | C7 | -0.0204 |
| C2 | -0.0268 | C8 | -0.0060 |
| C3 | -0.0059 | C9 | 0.0126 |
| C4 | 0.0135 | O1 | 0.0361 |
| C5 | 0.0117 | O3 | 0.0180 |
| C6 | 0.0033 | | |

Dihedral angle between planes (b) & (c) $45.5^{\circ}(2.0)^{\circ}$

5. Anisotropic thermal parameters for 2-phenylisatogen ($\times 10^4$) of the form: $\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}-U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\right]$ e.s.d.'s in parentheses.

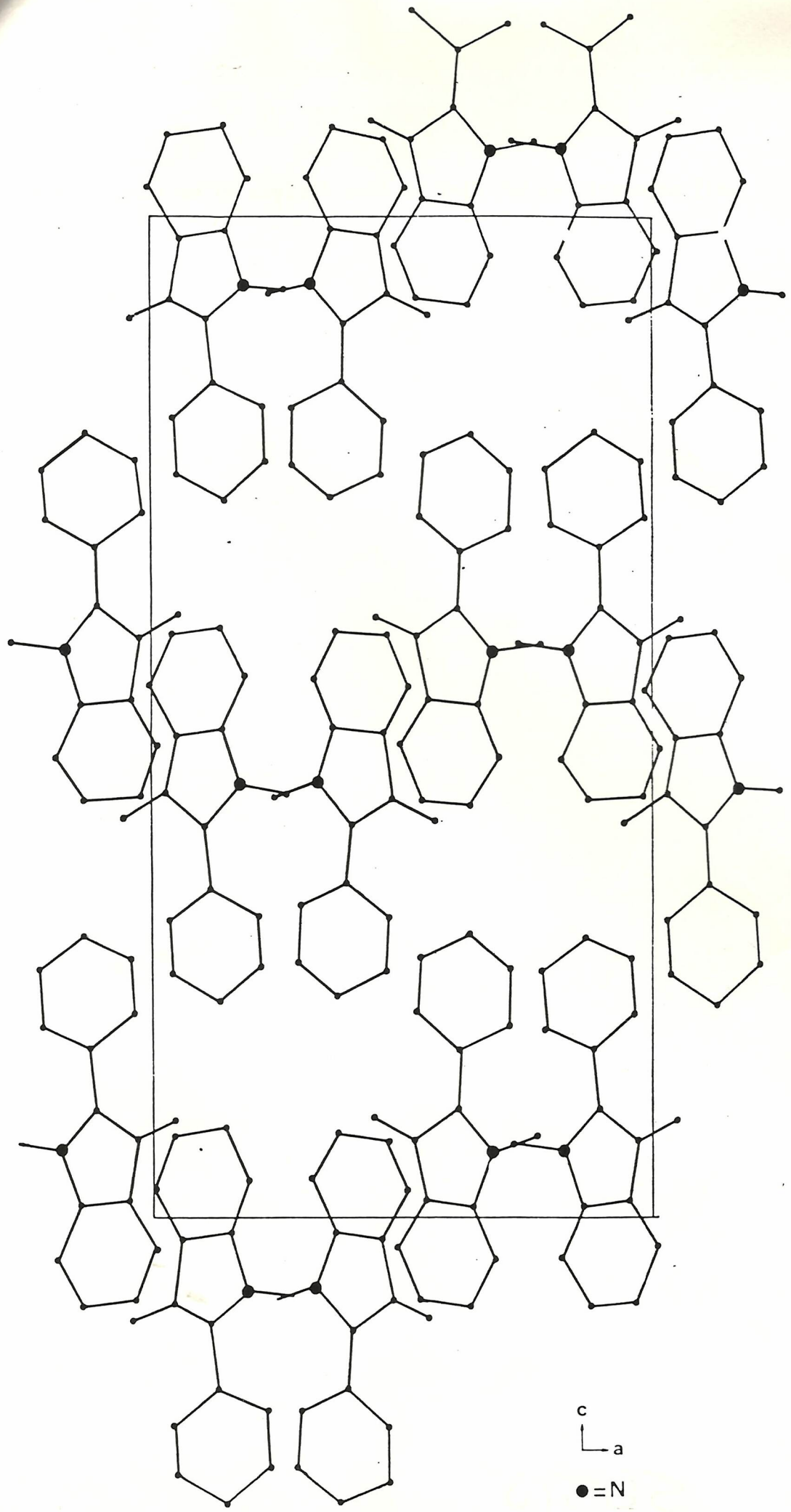
| ATOM | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| N(1) | 581 (12) | 491 (12) | 636 (14) | 03 (10) | 38 (11) | -14 (11) |
| C(2) | 437 (13) | 406 (12) | 512 (14) | -36 (11) | 80 (11) | -25 (12) |
| C(3) | 413 (12) | 475 (13) | 467 (14) | -46 (11) | 43 (11) | -27 (11) |
| C(4) | 493 (14) | 461 (14) | 506 (15) | -60 (12) | 10 (12) | -77 (12) |
| C(5) | 417 (14) | 456 (13) | 487 (14) | -19 (12) | 15 (12) | -93 (12) |
| C(6) | 624 (17) | 694 (19) | 626 (19) | -147 (15) | 134 (14) | -101 (15) |
| C(7) | 869 (21) | 807 (21) | 534 (18) | -132 (16) | 141 (16) | -228 (19) |
| C(8) | 836 (20) | 774 (19) | 515 (18) | 21 (15) | -90 (16) | -189 (18) |
| C(9) | 616 (16) | 594 (17) | 621 (18) | -07 (14) | -69 (14) | -68 (14) |
| C(10) | 563 (14) | 434 (13) | 467 (14) | 23 (11) | 30 (12) | -34 (12) |
| C(11) | 721 (20) | 715 (18) | 626 (19) | 46 (15) | 08 (14) | 192 (16) |
| C(12) | 985 (26) | 836 (21) | 659 (20) | 160 (16) | -98 (18) | 184 (20) |
| C(13) | 938 (22) | 813 (21) | 528 (18) | 111 (16) | 10 (16) | -45 (20) |
| C(14) | 740 (20) | 118 (25) | 524 (19) | 37 (17) | 87 (15) | -06 (21) |
| C(15) | 661 (12) | 849 (20) | 550 (17) | 16 (16) | 53 (14) | 86 (17) |
| O(1) | 666 (11) | 756 (13) | 747 (12) | 69 (11) | 130 (10) | 324 (11) |
| O(2) | 656 (11) | 833 (14) | 620 (12) | -30 (10) | 38 (9) | 276 (10) |

6. Anisotropic thermal parameters for 2-(2-pyridyl)isatogen ($\times 10^4$) of the form:

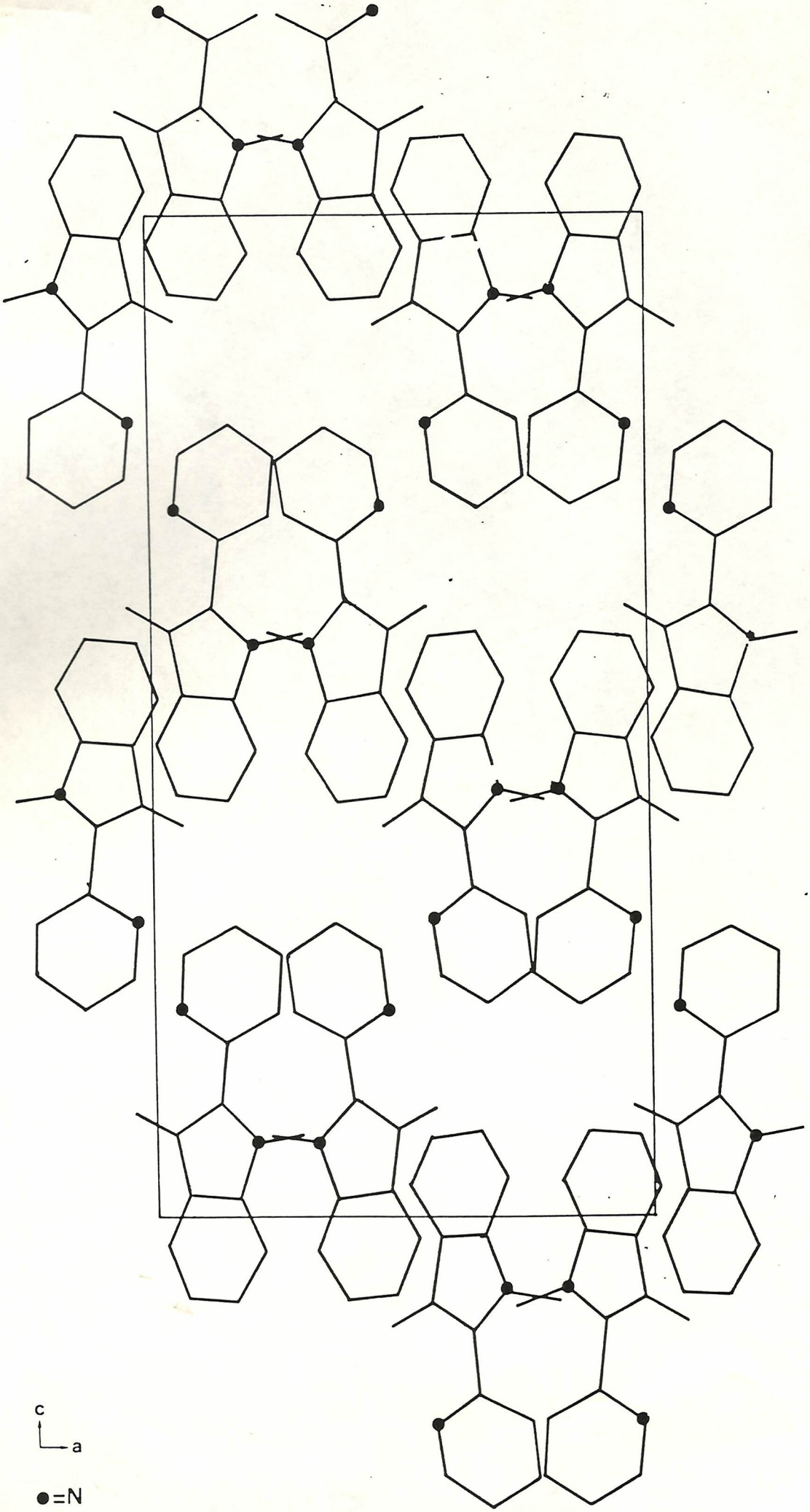
$$\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}-U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)\right]$$ e.s.d.'s in parentheses.

| ATOM | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| N(1) | 492 (19) | 473 (21) | 538 (21) | -14 (17) | -12 (16) | -12 (19) |
| C(2) | 420 (21) | 419 (24) | 416 (21) | 81 (20) | -82 (18) | -3 (21) |
| C(3) | 352 (19) | 444 (25) | 435 (21) | 59 (20) | -27 (18) | -59 (20) |
| C(4) | 415 (20) | 432 (25) | 423 (22) | 66 (20) | 04 (20) | -27 (20) |
| C(5) | 442 (21) | 431 (24) | 374 (22) | 21 (20) | 24 (20) | -85 (21) |
| C(6) | 542 (23) | 576 (29) | 442 (25) | 42 (21) | -90 (20) | -53 (23) |
| C(7) | 668 (28) | 666 (33) | 462 (25) | 53 (24) | -129 (24) | -118 (28) |
| C(8) | 749 (29) | 620 (30) | 402 (23) | -38 (23) | -32 (24) | -152 (28) |
| C(9) | 453 (22) | 582 (29) | 629 (29) | -38 (25) | -75 (21) | 11 (21) |
| O(1) | 522 (18) | 712 (20) | 636 (18) | -75 (16) | -104 (15) | 238 (15) |
| O(3) | 520 (15) | 855 (22) | 604 (17) | 92 (16) | -35 (15) | 182 (18) |
| C(10) | 459 (22) | 420 (24) | 425 (21) | -19 (20) | -48 (20) | 19 (21) |
| C(11) | 536 (24) | 597 (30) | 477 (24) | -1 (24) | -10 (21) | -45 (23) |
| C(12) | 649 (25) | 670 (32) | 541 (27) | -25 (26) | 110 (25) | -72 (25) |
| C(13) | 768 (30) | 625 (33) | 448 (23) | -40 (22) | 24 (25) | 121 (27) |
| C(14) | 637 (28) | 851 (36) | 500 (28) | -123 (24) | -130 (25) | 64 (28) |
| N(15) | 509 (19) | 733 (26) | 470 (19) | -63 (18) | -113 (17) | -47 (19) |

9. Structure of crystal cell 2-phenylisatogen (1a)


c
a
● = N

*10.* Structure of crystal cell of 2-(2-pyridyl)isatogen (1b)


c
a
●=N