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# Crystal Structure Determination of *N*-Phenyl-(5-anilino-2,4-pentadienylidene) ammonium Bromide Hemihydrate. A Test on the Applicability of a Relation between Structure Factor, Triple Products and a Single Patterson Vector

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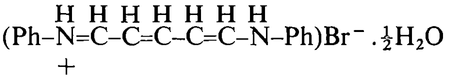
The crystal structure of one modification of *N*-phenyl-(5-anilino-2,4-pentadienylidene)ammonium bromide hemihydrate has been determined by the heavy-atom method. In order to test a procedure based on a relation between structure factor, triple products and a single Patterson vector, the positions of the bromine atoms were determined with the aid of this procedure. The crystals are monoclinic, space group $C2/c$, $Z=8$, with $a=14{\cdot}01$, $b=13{\cdot}92$, $c=16{\cdot}59$ Å, $\beta=96{\cdot}27°$. Intensities were measured on an automatic 4-circle diffractometer. A block-diagonal least-squares refinement led to a final $R$ of 0·064. The molecule is nearly planar and is in an all-*trans* configuration. There are no alternating C–C bond distances in the pentadienylidene chain which thus has aromatic character. The water molecule lies on a twofold axis and participates in four weak hydrogen bonds of the type O–H···Br⁻ and O···H–C in a pseudo-tetrahedral arrangement. The bromine ion is connected by hydrogen bonds to one water molecule and to two nitrogen atoms; this arrangement is nearly planar and is asymmetric.

## Introduction

Several modifications of the compound *N*-phenyl-(5-anilino-2,4-pentadienylidene)ammonium bromide hemihydrate,



$$(\mathrm{Ph{-}\underset{+}{\overset{H}{N}}{=}\overset{H}{C}{-}\overset{H}{C}{=}\overset{H}{C}{-}\overset{H}{C}{=}\overset{H}{C}{-}\overset{H}{N}{-}Ph)Br^{-}.\tfrac{1}{2}H_2O}$$

(hereinafter called: PAPAB), are known (König, 1904). Our first aim was to determine the crystal structure of a monoclinic modification of PAPAB, in order to establish the geometry of the molecule and the bonding character in the pentadienylidene chain. Secondly we wanted to test the applicability of a procedure to determine the heavy-atom positions, employing a relation between structure factor, triple products and a single (heavy-atom) Patterson vector (Krabbendam & Kroon, 1971).

## Experimental

A sample of PAPAB was kindly supplied by Professor J. F. Arens, who also suggested the X-ray analysis. The block-shaped crystals obtained by slow evaporation of a glacial acetic acid solution (König, 1904) show a blue metallic shine and are ruby-red in transmitted light. They apparently belong to the α modification described by Shimidzu (1927). Space group, cell constants and structure factors were deduced from measurements on a four-circle automatic Enraf–Nonius diffractometer with Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å).

### *Crystal data*

$\mathrm{[Ph{-}NH{-}(CH)_5{-}NH{-}Ph]Br.\tfrac{1}{2}H_2O}$, M.W. 338·24.
Monoclinic
$a=14{\cdot}01$, $b=13{\cdot}92$, $c=16{\cdot}59$ Å, $\beta=96{\cdot}27°$,
$V_{\text{cell}}=3218$ Å$^3$, $D_c=1{\cdot}39$ g cm$^{-3}$, $Z=8$. $F(000)=1384$.
Systematic absences: $hkl$ with $h+k=2n+1$, $h0l$ with $l=2n+1$.

The symmetry test of Ramachandran & Srinivasan (1959) revealed the presence of a centre of symmetry. Thus the space group is $C2/c$.

The $\omega$ scan mode was used for recording the intensities. A hemisphere of the reciprocal lattice was scanned, up to $\theta=70°$, and, after averaging, 2810 independent intensities were available for the structure determination. No correction for absorption ($\mu=36{\cdot}4$ cm$^{-1}$) was applied; the dimensions of the crystal used were 0·15 × 0·07 × 0·05 mm.

## Structure determination

In a preceding paper (Krabbendam & Kroon, 1971) we established a relation between a structure factor, triple products and a single Patterson vector **U** by comparing the Fourier coefficients of the structure with the Fourier coefficients of the Hoppe section at a single vector **U** in the double Patterson.

It reads:

$$F_{\mathbf{h}}=\frac{\sum_{\mathbf{h}'}F_{\mathbf{h}}F_{\mathbf{h}'}F_{\overline{\mathbf{h}+\mathbf{h}'}}\cos 2\pi\mathbf{h}'\,.\,\mathbf{U}}{\sum_{\mathbf{h}'}f^M_{\mathbf{h}}f^M_{\overline{\mathbf{h}+\mathbf{h}'}}\cos 2\pi\mathbf{h}\,.\,\mathbf{U}/2}\,, \tag{1}$$

in which **U** is a single vector between atoms $M$ with form factor $f^M_{\mathbf{h}}$. Employing absolute values of the triple products, each provided with a weighting factor $P_+-P_-$, this relation has been modified to:

$$F_{\mathbf{h}}=\frac{\sum_{\mathbf{h}'}|F_{\mathbf{h}}F_{\mathbf{h}'}F_{\overline{\mathbf{h}+\mathbf{h}'}}|\tanh N(|U_{\mathbf{h}}U_{\mathbf{h}'}U_{\overline{\mathbf{h}+\mathbf{h}'}}|)\cos 2\pi\mathbf{h}'\,.\,\mathbf{U}}{\sum_{\mathbf{h}'}f^M_{\mathbf{h}}f^M_{\overline{\mathbf{h}+\mathbf{h}'}}\cos 2\pi\mathbf{h}\,.\,\mathbf{U}/2}\,. \tag{2}$$

We expected to find the single vectors **U** by searching for those values of **U** which minimize the measure of fit $R_A$, defined as:

$$R_A = \frac{\sum_{\mathbf{h}} ||F^O_{\mathbf{h}}| - |F^C_{\mathbf{h}}||}{\sum_{\mathbf{h}} |F^O_{\mathbf{h}}|}, \tag{3}$$

in which $F^C_{\mathbf{h}}$ stands for the right-hand side of (2). However, in a test run on a two-dimensional example (plane group *pgg*), it became apparent that the function $R_A(\mathbf{U})$ showed minima at the positions of double peaks as well. We then made use of the fact that all hypersections of the double Patterson belong to plane group *p*2, with the exception of the hypersections at the single peaks, which retain the symmetry of the structure. This led to a measure of fit $R_B$, which required the Fourier coefficients of a hypersection to obey the symmetry relations of the plane group *pgg*. The function $R_B(\mathbf{U})$ also showed a few false minima in addition to the correct ones, but not on the positions of the double vectors, as was the case in $R_A(\mathbf{U})$. We devised a third measure of fit $R_C$, a combination of $R_A$ and $R_B$, which indeed showed its lowest minima at the positions of the single vectors.

We have employed equation (2), together with $R_A$, $R_B$ and $R_C$, to determine the structure of a derivative of chloranilic acid of unknown chemical composition, space group $C2/c$ (Kroon & Krabbendam, 1973). For this space group $R_B$ and $R_C$ were defined as:

$$R_B = \frac{\sum_h \sum_k \sum_l [|F^C_{hkl} - (-1)^l F^C_{h\bar{k}l}|]}{\sum_h \sum_k \sum_l |F^C_{hkl}|} \tag{4}$$

$$R_C = \frac{\sum_h \sum_k \sum_l ||F^O_{hkl}| - [|F^C_{hkl} + (-1)^l F^C_{h\bar{k}l}| \cdot \frac{1}{2}]|}{\sum_h \sum_k \sum_l |F^O_{hkl}|} . \tag{5}$$

Searching for a chlorine–chlorine single vector, we scanned **U** at regular intervals through Patterson space, found a minimum in $R_C(\mathbf{U})$, calculated the signs of $F_{\mathbf{h}}$ from the triple products by equation (2), and derived from the resulting $F_{\text{obs}}$ map the structure (which appeared to be potassium chloranilate monohydrate).

As the scanning procedure was rather time-consuming, we wanted to test a simpler procedure on the structure determination of PAPAB: The coordinates of the 18 highest Patterson peaks were introduced into equation (2), using the bromine scattering factors; $R_A$, $R_B$ and $R_C$ were calculated in each case. Owing to the limited size of the computer storage only 684 equations, each with 1007 terms, were employed. The result is shown in Table 1. In this table each search vector $(u,v,w)$ goes with a vector $(\frac{1}{2}-u, \frac{1}{2}-v, -w)$. The reason for this is that the centres of symmetry in $C2/c$ belong to one of two different equivalence classes, so that for each single vector $(u,v,w)$, which connects two atoms related by a centre of symmetry in one equivalence class, there is a second vector $(\frac{1}{2}-u, \frac{1}{2}-v, -w)$, which connects two atoms related by a centre of symmetry in the other equivalence class. The symmetry relations between structure factors, and consequently the expressions for $R_B$ and $R_C$, depend on the equivalence class; once these expressions are chosen only one of a pair of possible single vectors will give a minimum in $R_B$ and $R_C$, but we have to check both vectors of each pair. If we take this into consideration and also disregard the rather special Harker vector at (0·0000, 0·3889, 0·5000) then we find, from Table 1, for the lowest value of $R_B$ ($100R_B = 43$) and of $R_C$ ($100R_C = 35$) the bromine–bromine single vector at $\mathbf{U} = (0{\cdot}1667, 0{\cdot}3889, 0{\cdot}2639)$. To investigate the influence of overlap from neighbouring hypersections in the Hoppe section, we applied a sharpening function $\exp 5s^2$ to the $F$'s and $f^{\mathrm{Br}}$'s in equation (2). As a result there was a considerable decrease in the $R_A$ values for all search vectors, whereas $R_B$ showed a significant decrease only at the position of the single vector.

Table 1. *The measures of fit $R_A$, $R_B$ and $R_C$ as a function of the Patterson vectors* $(u,v,w)$

For each $R$ the first column is calculated with unsharpened, the sccond with sharpened reflexions. $u$, $v$ and $w$ are $\times 10^4$.

| $u$ | $v$ | $w$ | $100R_A$ | | $100R_B$ | | $100R_C$ | | |
|---|---|---|---|---|---|---|---|---|---|
| 5000 | 1111 | 5000 | 23 | 13 | 121 | 122 | 56 | 53 | |
| 0000 | 3889 | 5000 | 23 | 13 | 0 | 0 | 24 | 13 | |
| 3277 | 5000 | 2388 | 24 | 14 | 114 | 112 | 54 | 50 | |
| 1723 | 0000 | 7612 | 24 | 14 | 108 | 107 | 52 | 48 | |
| 3333 | 1111 | 7361 | 33 | 26 | 127 | 130 | 55 | 53 | |
| 1667 | 3889 | 2639 | 33 | 26 | 43 | 33 | 35 | 26 | single vector |
| 3236 | 3500 | 9055 | 57 | 57 | 88 | 86 | 64 | 65 | |
| 1764 | 1500 | 0945 | 57 | 57 | 130 | 126 | 71 | 72 | |
| 4527 | 3263 | 8263 | 62 | 62 | 110 | 112 | 71 | 73 | |
| 0473 | 1737 | 1737 | 62 | 62 | 128 | 127 | 74 | 75 | |
| 3650 | 3150 | 8477 | 65 | 67 | 115 | 111 | 74 | 77 | |
| 1350 | 1850 | 1523 | 65 | 67 | 129 | 128 | 75 | 79 | |
| 2819 | 4250 | 8055 | 68 | 66 | 91 | 90 | 74 | 74 | |
| 2181 | 0750 | 1945 | 68 | 66 | 129 | 121 | 78 | 78 | |
| 2388 | 3444 | 9361 | 64 | 63 | 103 | 105 | 72 | 73 | |
| 2612 | 1556 | 0639 | 64 | 63 | 135 | 126 | 76 | 76 | |
| 3416 | 2638 | 9722 | 71 | 74 | 138 | 127 | 80 | 83 | |
| 1584 | 2362 | 0278 | 71 | 74 | 126 | 125 | 78 | 83 | |
| 0153 | 5000 | 8277 | 64 | 66 | 116 | 113 | 79 | 81 | |
| 4847 | 0000 | 1723 | 64 | 66 | 116 | 113 | 79 | 81 | |
| 1805 | 5000 | 5944 | 65 | 64 | 108 | 109 | 79 | 78 | |
| 3195 | 0000 | 4056 | 65 | 64 | 103 | 105 | 78 | 78 | |
| 4888 | 2819 | 7583 | 62 | 64 | 133 | 126 | 71 | 74 | |
| 0112 | 2181 | 2417 | 62 | 64 | 134 | 137 | 71 | 75 | |
| 1722 | 2555 | 6250 | 63 | 62 | 130 | 133 | 75 | 74 | |
| 3278 | 2445 | 3750 | 63 | 62 | 132 | 130 | 74 | 74 | |
| 3819 | 4888 | 0833 | 67 | 76 | 110 | 120 | 76 | 84 | |
| 1181 | 0112 | 9167 | 67 | 76 | 125 | 126 | 77 | 84 | |
| 0777 | 4305 | 3861 | 65 | 68 | 112 | 120 | 74 | 79 | |
| 4223 | 0695 | 6139 | 65 | 68 | 114 | 118 | 74 | 78 | |
| 2014 | 2916 | 0583 | 75 | 78 | 119 | 116 | 81 | 84 | |
| 2986 | 2084 | 9417 | 75 | 78 | 127 | 130 | 82 | 86 | |
| 3805 | 5000 | 4666 | 76 | 73 | 113 | 113 | 85 | 85 | |
| 1195 | 0000 | 5334 | 76 | 73 | 96 | 95 | 84 | 83 | |
| 2847 | 2708 | 5556 | 68 | 72 | 126 | 131 | 77 | 82 | |
| 2153 | 2292 | 4444 | 68 | 72 | 122 | 125 | 77 | 81 | |

It appears that $R_B(\mathbf{U})$ is an excellent criterion to identify the heavy-atom single vector. However, care must be taken to avoid special vectors such as, in the present case, the Harker vector at (0·0000, 0·3889, 0·5000). It is not difficult to show that, in general, Harker vectors that originate from glide-plane operations correspond to hypersections with the exact space-group symmetry (that is: with $R_B=0$). These special vectors are known for each space group and can be excluded from the analysis.

A heavy-atom map, with all observed structure factors, and signs calculated from the bromine positions at $\frac{1}{2}\mathbf{U}$, revealed the structure at once. Another Fourier map, with signs obtained directly from the triple products by application of equation (2), was inferior to the first. The $F_{\text{obs}}$ Fourier map in the structure determination of potassium chloranilate monohydrate, with signs calculated from the triple products, had revealed the structure very clearly. The inferiority of the Fourier map in the present case is due to the small number of reflexions. This follows from the fact that the signs obtained by (2), for this small number of structure factors, agree remarkably well with the signs obtained from the heavy-atom positions.

Another problem in the calculation of $F_{\mathbf{h}}^C$, and thus of $R_C$, is the fact that sometimes $\cos 2\pi\mathbf{h}\,.\,\mathbf{U}/2$ in the denominator of (1) and (2) is close to zero. In this case equation (1), and equation (2) correspondingly, should be replaced by the equivalent relation:

$$F_{\mathbf{h}}=\frac{-\sum_{\mathbf{h}'}F_{\mathbf{h}}F_{\mathbf{h}'}F_{\overline{\mathbf{h}+\mathbf{h}'}}\sin 2\pi\mathbf{h}'\,.\,\mathbf{U}}{\sum_{\mathbf{h}'}f_{\mathbf{h}'}^{M}f_{\overline{\mathbf{h}+\mathbf{h}'}}^{M}\sin 2\pi\mathbf{h}\,.\,\mathbf{U}/2}\,. \tag{6}$$

We have not done this yet; we merely left out $F_{\mathbf{h}}^C$ in the calculation of $R_B$ and $R_C$ if this happened.

However, our main concern is with the relatively few reflexions we could handle in our computer program. Since the relations (1) and (6) are algebraic, they are valid only if all structure factors are included. We do not plan to make the program handle more reflexions. Instead, we will proceed by employing a recently derived statistical relation, based on the joint probability distribution $P(E_{\mathbf{k}},E_{\mathbf{h}-\mathbf{k}},\cos 2\pi\mathbf{k}\,.\,\mathbf{U})$, which involves a conditional average over only the larger $E$ values (Heinerman, Krabbendam & Kroon, 1974).

Table 2. *Fractional coordinates and thermal parameters*

Estimated standard deviations in parentheses. The anisotropic temperature factor is in the form $\exp[10^{-5}\sum_i\sum_j h_ih_j\beta_{ij}]$.

| | $x$ | $y$ | $z$ | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $2\beta_{12}$ | $2\beta_{23}$ | $2\beta_{31}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br | 0·08158 (4) | 0·19648 (4) | 0·13182 (4) | 467 | 506 | 394 | 149 | 161 | 141 |
| O | 0·0000 | 0·3683 (4) | 0·2500 | 567 | 535 | 510 | 0 | 0 | 67 |
| N(1) | 0·6007 (3) | 0·1708 (3) | −0·0394 (3) | 377 | 491 | 333 | 62 | 156 | 119 |
| N(2) | 0·2469 (3) | 0·0448 (3) | 0·2259 (3) | 382 | 410 | 373 | 88 | 84 | 148 |
| C(1) | 0·5702 (4) | 0·1212 (4) | 0·0207 (3) | 463 | 440 | 312 | −22 | 40 | 95 |
| C(2) | 0·4811 (4) | 0·1320 (4) | 0·0446 (3) | 400 | 510 | 379 | 92 | 99 | 179 |
| C(3) | 0·4486 (4) | 0·0800 (4) | 0·1084 (3) | 392 | 524 | 394 | 118 | 23 | 183 |
| C(4) | 0·3585 (4) | 0·0895 (4) | 0·1341 (3) | 484 | 442 | 401 | 100 | 102 | 236 |
| C(5) | 0·3318 (4) | 0·0358 (4) | 0·1973 (3) | 396 | 431 | 387 | −26 | 86 | 131 |
| C(6) | 0·6923 (3) | 0·1677 (3) | −0·0684 (3) | 417 | 381 | 308 | −8 | −57 | 154 |
| C(7) | 0·7667 (4) | 0·1126 (4) | −0·0314 (3) | 448 | 608 | 337 | 234 | 59 | 129 |
| C(8) | 0·8541 (4) | 0·1129 (5) | −0·0636 (4) | 472 | 714 | 457 | 188 | 149 | 198 |
| C(9) | 0·8670 (4) | 0·1659 (4) | −0·1322 (4) | 523 | 653 | 473 | −92 | −95 | 366 |
| C(10) | 0·7917 (4) | 0·2210 (4) | −0·1684 (3) | 571 | 578 | 400 | −82 | 84 | 264 |
| C(11) | 0·7039 (4) | 0·2210 (4) | −0·1364 (3) | 473 | 454 | 369 | −66 | 119 | 103 |
| C(12) | 0·2137 (3) | −0·0032 (3) | 0·2925 (3) | 416 | 355 | 314 | −2 | −55 | 121 |
| C(13) | 0·1238 (3) | 0·0228 (4) | 0·3124 (3) | 379 | 457 | 375 | −25 | 91 | 168 |
| C(14) | 0·0867 (4) | −0·0227 (4) | 0·3756 (4) | 422 | 549 | 449 | −68 | 33 | 251 |
| C(15) | 0·1375 (4) | −0·0942 (4) | 0·4192 (3) | 629 | 497 | 373 | −150 | 74 | 321 |
| C(16) | 0·2264 (4) | −0·1199 (4) | 0·3984 (3) | 650 | 482 | 382 | 130 | 186 | 213 |
| C(17) | 0·2657 (4) | −0·0749 (4) | 0·3349 (3) | 486 | 444 | 383 | 101 | 42 | 155 |

Table 2 (*cont.*)

| | $x$ | $y$ | $z$ | $B$(Å$^2$) | Bonded to |
|---|---|---|---|---|---|
| H(1) | 0·005 | 0·330 | 0·210 | 5 | O |
| H(2) | 0·560 | 0·215 | −0·075 | 4 | N(1) |
| H(3) | 0·205 | 0·095 | 0·207 | 4 | N(2) |
| H(4) | 0·617 | 0·075 | 0·057 | 4 | C(1) |
| H(5) | 0·433 | 0·174 | 0·020 | 4 | C(2) |
| H(6) | 0·490 | 0·025 | 0·127 | 4 | C(3) |
| H(7) | 0·305 | 0·133 | 0·105 | 4 | C(4) |
| H(8) | 0·375 | −0·010 | 0·228 | 4 | C(5) |
| H(9) | 0·765 | 0·093 | 0·027 | 4 | C(7) |
| H(10) | 0·910 | 0·073 | −0·025 | 4 | C(8) |
| H(11) | 0·930 | 0·170 | −0·170 | 4 | C(9) |
| H(12) | 0·793 | 0·270 | −0·235 | 4 | C(10) |
| H(13) | 0·655 | 0·272 | −0·165 | 4 | C(11) |
| H(14) | 0·085 | 0·058 | 0·275 | 4 | C(13) |
| H(15) | 0·020 | 0·000 | 0·393 | 4 | C(14) |
| H(16) | 0·107 | −0·133 | 0·465 | 4 | C(15) |
| H(17) | 0·265 | −0·165 | 0·437 | 4 | C(16) |
| H(18) | 0·328 | −0·105 | 0·313 | 4 | C(17) |

## Structure refinement

The atomic coordinates, deduced from the heavy-atom Fourier synthesis, were refined by block-diagonal least-squares calculations. The hydrogen-atom positions were found from a difference synthesis. They were given a $B$ of 4 Å$^2$, except for the water-molecule hydrogen atoms, which were assigned a $B$ of 5 Å$^2$. These values were kept fixed during further block-diagonal least-squares refinement, in which all positional parameters and the anisotropic thermal parameters of the non-hydrogen atoms were varied. The scattering factors for the heavy atoms were obtained from Cromer & Mann (1968); for hydrogen the analytical form of

Table 3. *Observed and calculated structure factors*

The entries of each column are $h,k,l,\lvert F^{\mathrm{obs}}\rvert,\lvert F^{\mathrm{calc}}\rvert$ respectively.

| | | | | |
|---|---|---|---|---|
| 0 | 0 | 2 | 6 | 6 |
| | | 4 | 282 | 286 |
| | | 6 | 44 | 39 |
| | | 8 | 123 | 128 |
| | | 10 | 74 | 73 |
| | | 12 | 124 | 125 |
| | | 14 | 28 | 31 |
| | | 16 | 40 | 40 |
| | | 18 | 27 | 34 |
| | | 20 | 24 | 24 |
| 2 | 0 | -20 | 5 | 8 |
| | | -18 | 11 | 12 |
| | | -16 | 58 | 57 |
| | | -14 | 24 | 29 |
| | | -12 | 101 | 104 |
| | | -10 | 45 | 49 |
| | | -8 | 222 | 215 |
| | | -6 | 174 | 169 |
| | | -4 | 102 | 107 |
| | | -2 | 139 | 139 |
| | | 0 | 66 | 67 |
| | | 2 | 60 | 64 |
| | | 4 | 35 | 38 |
| | | 6 | 146 | 151 |
| | | 8 | 39 | 36 |
| | | 10 | 112 | 114 |
| | | 14 | 17 | 20 |
| | | 16 | 7 | 9 |
| | | 18 | 38 | 39 |
| 4 | 0 | -20 | 8 | 5 |
| | | -18 | 59 | 60 |
| | | -16 | 23 | 19 |
| | | -14 | 88 | 91 |
| | | -12 | 18 | 9 |
| | | -10 | 115 | 109 |
| | | -8 | 31 | 31 |
| | | -6 | 179 | 174 |
| | | -4 | 118 | 114 |
| | | -2 | 119 | 121 |
| | | 0 | 87 | 83 |
| | | 2 | 226 | 232 |
| | | 4 | 194 | 200 |
| | | 6 | 84 | 84 |
| | | 8 | 43 | 45 |
| | | 10 | 65 | 67 |
| | | 12 | 76 | 79 |
| | | 14 | 11 | 16 |
| | | 16 | 83 | 91 |
| 6 | 0 | -18 | 20 | 22 |
| | | -16 | 10 | 13 |
| | | -12 | 105 | 101 |
| | | -10 | 16 | 19 |
| | | -8 | 107 | 103 |
| | | -6 | 37 | 36 |
| | | -4 | 68 | 78 |
| | | -2 | 33 | 33 |
| | | 0 | 221 | 221 |
| | | 4 | 187 | 171 |
| | | 6 | 86 | 86 |
| | | 8 | 168 | 170 |
| | | 10 | 37 | 36 |
| | | 12 | 50 | 53 |
| | | 14 | 19 | 26 |
| | | 18 | 14 | 6 |
| 8 | 0 | -16 | 40 | 40 |
| | | -14 | 7 | 6 |
| | | -12 | 49 | 50 |
| | | -10 | 58 | 54 |
| | | -8 | 113 | 112 |
| | | -6 | 47 | 49 |
| | | -4 | 140 | 139 |
| | | -2 | 127 | 128 |
| | | 0 | 23 | 24 |
| | | 2 | 89 | 90 |
| | | 4 | 51 | 49 |
| | | 6 | 94 | 97 |
| | | 8 | 10 | 8 |
| | | 10 | 33 | 37 |
| | | 12 | 15 | 13 |
| | | 14 | 36 | 33 |
| | | 16 | 12 | 5 |
| 10 | 0 | -16 | 13 | 9 |
| | | -14 | 41 | 39 |
| | | -12 | 17 | 14 |
| | | -10 | 93 | 92 |
| | | -6 | 94 | 92 |
| | | -4 | 27 | 27 |
| | | -2 | 99 | 97 |
| | | 0 | 38 | 40 |
| | | 2 | 85 | 86 |
| | | 4 | 29 | 31 |
| | | 6 | 46 | 45 |
| | | 10 | 27 | 25 |
| | | 12 | 31 | 28 |
| | | 14 | 18 | 17 |
| 12 | 0 | -14 | 19 | 23 |
| | | -12 | 29 | 32 |
| | | -10 | 39 | 42 |
| | | -8 | 31 | 29 |
| | | -6 | 11 | 19 |
| | | -4 | 64 | 62 |
| | | -2 | 36 | 35 |
| | | 0 | 67 | 71 |
| | | 2 | 20 | 20 |
| | | 4 | 13 | 17 |
| | | 6 | 33 | 34 |
| | | 8 | 75 | 79 |
| | | 10 | 14 | 12 |
| | | 12 | 31 | 32 |
| 14 | 0 | -12 | 33 | 31 |
| | | -10 | 13 | 5 |
| | | -8 | 44 | 48 |
| | | -6 | 29 | 25 |
| | | -4 | 56 | 61 |
| | | -2 | 48 | 49 |
| | | 0 | 57 | 54 |
| | | 2 | 49 | 48 |
| | | 4 | 34 | 33 |
| | | 6 | 37 | 38 |
| | | 8 | 21 | 19 |
| 16 | 0 | -8 | 5 | 3 |
| | | -6 | 38 | 39 |
| | | -4 | 10 | 10 |
| | | -2 | 30 | 27 |
| | | 0 | 10 | 10 |
| | | 2 | 34 | 33 |
| | | 4 | 19 | 19 |
| 1 | 1 | -20 | 18 | 19 |
| | | -19 | 12 | 2 |
| | | -18 | 7 | 3 |
| | | -17 | 36 | 34 |
| | | -16 | 24 | 22 |
| | | -15 | 35 | 32 |
| | | -14 | 13 | 6 |
| | | -13 | 58 | 60 |
| | | -12 | 55 | 52 |
| | | -11 | 30 | 32 |
| | | -10 | 62 | 60 |
| | | -9 | 25 | 29 |
| | | -8 | 39 | 41 |
| | | -7 | 113 | 114 |
| | | -6 | 16 | 15 |
| | | -5 | 35 | 42 |
| | | -4 | 88 | 86 |
| | | -3 | 140 | 144 |
| | | -2 | 6 | 2 |
| | | -1 | 151 | 153 |
| | | 0 | 27 | 28 |
| | | 1 | 102 | 99 |
| | | 2 | 89 | 88 |
| | | 3 | 86 | 83 |
| | | 4 | 152 | 149 |
| | | 5 | 195 | 196 |
| | | 7 | 26 | 26 |
| | | 9 | 165 | 165 |
| | | 10 | 51 | 49 |
| | | 12 | 19 | 19 |
| | | 13 | 72 | 71 |
| | | 14 | 38 | 41 |
| | | 16 | 15 | 16 |
| | | 17 | 33 | 34 |
| | | 18 | 16 | 16 |
| | | 19 | 6 | 15 |
| 3 | 1 | -19 | 33 | 33 |
| | | -18 | 7 | 10 |
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| | | -6 | 70 | 68 |
| | | -5 | 10 | 10 |
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| | | -3 | 163 | 164 |
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| | | -1 | 96 | 99 |
| | | 0 | 42 | 38 |
| | | 1 | 273 | 273 |
| | | 2 | 73 | 73 |
| | | 3 | 99 | 89 |
| | | 4 | 133 | 130 |
| | | 5 | 109 | 110 |
| | | 6 | 45 | 45 |
| | | 7 | 44 | 46 |
| | | 8 | 6 | 2 |
| | | 9 | 75 | 80 |
| | | 10 | 7 | 2 |
| | | 11 | 69 | 69 |
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| | | 4 | 24 | 23 |
| | | 5 | 53 | 50 |
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| | | 7 | 166 | 164 |
| | | 8 | 21 | 23 |
| | | 9 | 22 | 18 |
| | | 10 | 17 | 17 |
| | | 11 | 60 | 61 |
| | | 12 | 26 | 21 |
| | | 13 | 11 | 7 |
| | | 14 | 42 | 45 |
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| | | 16 | 13 | 10 |
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| | | 5 | 97 | 98 |
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| | | -12 | 22 | 22 |
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| | | -10 | 45 | 44 |
| | | -9 | 61 | 61 |
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| | | -7 | 110 | 109 |
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| | | -2 | 19 | 16 |
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| | | 7 | 33 | 32 |
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| | | 9 | 59 | 58 |
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| | | 0 | 42 | 42 |
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| | | 2 | 14 | 12 |
| | | 3 | 60 | 60 |
| | | 4 | 60 | 58 |
| | | 5 | 7 | 6 |
| | | 6 | 13 | 10 |
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| | | -7 | 7 | 7 |
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| | | 0 | 7 | 6 |
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| | | 3 | 74 | 76 |
| | | 4 | 43 | 43 |
| | | 5 | 58 | 57 |
| | | 6 | 32 | 32 |
| | | 7 | 36 | 37 |
| | | 8 | 12 | 2 |
| | | 9 | 36 | 35 |
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| | | 11 | 15 | 10 |
| 14 | 4 | -12 | 11 | 12 |
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| | | -10 | 10 | 7 |
| | | -9 | 9 | 10 |
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| | | 2 | 12 | 14 |
| | | 3 | 12 | 10 |
| | | 4 | 9 | 3 |
| | | 5 | 14 | 11 |
| | | 6 | 8 | 12 |
| | | 7 | 16 | 16 |
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| | | -11 | 12 | 14 |
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| | | -2 | 81 | 86 |
| | | -1 | 83 | 88 |
| | | 0 | 213 | 224 |
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| | | 2 | 180 | 179 |
| | | 3 | 5 | 3 |
| | | 4 | 164 | 161 |
| | | 5 | 32 | 34 |
| | | 6 | 149 | 150 |
| | | 7 | 8 | 9 |
| | | 8 | 106 | 106 |
| | | 9 | 6 | 6 |
| | | 10 | 53 | 50 |
| | | 11 | 24 | 27 |
| | | 12 | 46 | 47 |
| | | 13 | 15 | 14 |
| | | 14 | 22 | 22 |
| | | 15 | 15 | 13 |
| | | 16 | 18 | 23 |
| | | 18 | 26 | 30 |
| | | 19 | 5 | 4 |
| 3 | 5 | -19 | 10 | 3 |
| | | -18 | 12 | 13 |
| | | -17 | 7 | 6 |
| | | -16 | 40 | 43 |
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| | | -14 | 41 | 44 |
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| | | -6 | 122 | 128 |
| | | -5 | 89 | 81 |
| | | -4 | 26 | 24 |
| | | -2 | 179 | 181 |
| | | -1 | 55 | 51 |
| | | 0 | 50 | 50 |
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| | | 2 | 34 | 39 |
| | | 3 | 29 | 27 |
| | | 4 | 7 | 5 |
| | | 5 | 8 | 6 |
| | | 6 | 6 | 11 |
| | | 7 | 73 | 71 |
| | | 8 | 96 | 95 |
| | | 9 | 26 | 22 |
| | | 10 | 108 | 109 |
| | | 11 | 7 | 2 |
| | | 12 | 32 | 32 |
| | | 13 | 18 | 15 |
| | | 14 | 35 | 35 |
| | | 15 | 7 | 0 |
| | | 16 | 37 | 36 |
| | | 17 | 6 | 9 |
| | | 18 | 18 | 17 |
| 5 | 5 | -18 | 42 | 45 |
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| | | -16 | 26 | 26 |
| | | -15 | 14 | 13 |
| | | -14 | 54 | 54 |
| | | -13 | 24 | 22 |
| | | -12 | 35 | 36 |
| | | -11 | 14 | 14 |
| | | -10 | 40 | 40 |
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| | | -6 | 107 | 106 |
| | | -5 | 45 | 45 |
| | | -4 | 125 | 125 |
| | | -3 | 57 | 54 |
| | | -2 | 76 | 81 |
| | | -1 | 9 | 6 |
| | | 0 | 137 | 138 |
| | | 1 | 32 | 30 |
| | | 2 | 87 | 89 |
| | | 3 | 50 | 56 |
| | | 4 | 185 | 178 |
| | | 5 | 27 | 26 |
| | | 6 | 37 | 37 |
| | | 7 | 13 | 11 |
| | | 8 | 134 | 132 |
| | | 9 | 32 | 33 |
| | | 10 | 13 | 9 |
| | | 11 | 23 | 24 |
| | | 12 | 70 | 71 |
| | | 13 | 10 | 1 |
| | | 14 | 14 | 11 |
| | | 15 | 25 | 25 |
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| | | 17 | 16 | 10 |
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| | | -13 | 17 | 12 |
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| | | -10 | 30 | 26 |
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| | | -7 | 13 | 15 |
| | | -6 | 28 | 27 |
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| 7 | 5 | 2 | 73 | 70 |
| | | 3 | 27 | 28 |
| | | 4 | 97 | 98 |
| | | 5 | 17 | 19 |
| | | 6 | 60 | 63 |
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| | | 10 | 30 | 37 |
| | | 11 | 26 | 26 |
| | | 12 | 19 | 19 |
| | | 13 | 18 | 18 |
| | | 14 | 31 | 31 |
| | | 15 | 6 | 1 |
| | | 16 | 17 | 16 |
| 9 | 5 | -16 | 11 | 13 |
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| | | -13 | 18 | 16 |
| | | -12 | 41 | 39 |
| | | -11 | 26 | 27 |
| | | -10 | 62 | 62 |
| | | -9 | 32 | 32 |
| | | -8 | 91 | 90 |
| | | -7 | 18 | 21 |
| | | -6 | 74 | 76 |
| | | -5 | 17 | 16 |
| | | -4 | 18 | 17 |
| | | -3 | 47 | 49 |
| | | -2 | 154 | 157 |
| | | -1 | 26 | 26 |
| | | 0 | 10 | 8 |
| | | 1 | 22 | 22 |
| | | 2 | 76 | 73 |
| | | 3 | 22 | 22 |
| | | 4 | 12 | 14 |
| | | 5 | 30 | 28 |
| | | 6 | 57 | 54 |
| | | 7 | 11 | 9 |
| | | 8 | 17 | 18 |
| | | 9 | 19 | 10 |
| | | 10 | 49 | 52 |
| | | 11 | 7 | 0 |
| | | 12 | 13 | 12 |
| | | 13 | 6 | 1 |
| | | 14 | 23 | 21 |
| 11 | 5 | -14 | 32 | 30 |
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| | | -12 | 23 | 21 |
| | | -11 | 7 | 3 |
| | | -10 | 49 | 49 |
| | | -9 | 18 | 5 |
| | | -8 | 47 | 46 |
| | | -7 | 11 | 11 |
| | | -6 | 15 | 14 |
| | | -5 | 22 | 21 |
| | | -4 | 27 | 28 |
| | | -3 | 18 | 22 |
| | | -2 | 27 | 23 |
| | | -1 | 16 | 18 |
| | | 0 | 54 | 53 |
| | | 1 | 39 | 36 |
| | | 2 | 47 | 46 |
| | | 3 | 15 | 3 |
| | | 4 | 67 | 65 |
| | | 5 | 26 | 25 |
| | | 6 | 7 | 18 |
| | | 8 | 43 | 41 |
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| | | 11 | 13 | 2 |
| | | 12 | 18 | 16 |
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| | | -9 | 11 | 7 |
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| | | -3 | 18 | 24 |
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| | | -1 | 15 | 7 |
| | | 0 | 56 | 53 |
| | | 2 | 32 | 30 |
| | | 3 | 10 | 8 |
| | | 4 | 29 | 31 |
| | | 5 | 29 | 25 |
| | | 6 | 19 | 20 |
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| | | 8 | 39 | 39 |
| 15 | 5 | -8 | 20 | 19 |
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| | | -4 | 9 | 8 |
| | | -2 | 35 | 37 |
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| | | 4 | 11 | 10 |
| | | 5 | 18 | 4 |
| 0 | 6 | 0 | 79 | 80 |
| | | 1 | 170 | 166 |
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| | | 3 | 39 | 37 |
| | | 4 | 16 | 19 |
| | | 5 | 48 | 50 |
| | | 7 | 79 | 80 |
| | | 8 | 26 | 26 |
| | | 9 | 47 | 44 |
| | | 10 | 10 | 8 |
| | | 11 | 26 | 22 |
| | | 12 | 63 | 63 |
| | | 13 | 55 | 52 |
| | | 14 | 15 | 12 |
| | | 15 | 10 | 8 |
| | | 16 | 27 | 28 |
| | | 17 | 34 | 30 |
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| | | -17 | 20 | 21 |
| | | -16 | 19 | 17 |
| | | -15 | 46 | 50 |
| | | -14 | 22 | 22 |
| | | -13 | 25 | 26 |
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| | | -6 | 99 | 93 |
| | | -5 | 22 | 18 |
| | | -4 | 53 | 52 |
| | | -3 | 95 | 95 |
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| | | 0 | 80 | 80 |
| | | 1 | 130 | 130 |
| | | 2 | 67 | 66 |
| | | 3 | 67 | 66 |
| | | 4 | 33 | 33 |
| | | 5 | 216 | 219 |
| | | 6 | 157 | 155 |
| | | 7 | 54 | 55 |
| | | 8 | 41 | 40 |
| | | 9 | 66 | 66 |
| | | 10 | 74 | 73 |
| | | 11 | 108 | 109 |
| | | 12 | 7 | 5 |
| | | 13 | 38 | 31 |
| | | 14 | 7 | 6 |
| | | 15 | 37 | 36 |
| | | 16 | 9 | 8 |
| | | 17 | 21 | 24 |
| | | 18 | 18 | 15 |
| 4 | 6 | -18 | 11 | 9 |
| | | -17 | 22 | 25 |
| | | -16 | 10 | 1 |
| | | -15 | 29 | 24 |
| | | -13 | 49 | 52 |
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| | | -11 | 56 | 55 |
| | | -10 | 24 | 23 |
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| | | -6 | 68 | 70 |
| | | -5 | 128 | 131 |
| | | -4 | 6 | 8 |
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| | | 2 | 78 | 68 |
| | | 3 | 185 | 183 |

Table 3 (*cont.*)

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| | | 10 | 7 | 2 |
| | | 11 | 42 | 37 |
| | | 12 | 21 | 21 |
| | | 13 | 20 | 18 |
| | | 14 | 32 | 32 |
| | | 15 | 38 | 36 |
| | | 16 | 11 | 7 |
| | | 17 | 14 | 13 |
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| | | -7 | 50 | 51 |
| | | -6 | 11 | 12 |
| | | -5 | 65 | 70 |
| | | -4 | 67 | 70 |
| | | -3 | 66 | 66 |
| | | -2 | 28 | 26 |
| | | -1 | 74 | 79 |
| | | 0 | 48 | 51 |
| | | 1 | 71 | 72 |
| | | 2 | 12 | 8 |
| | | 3 | 66 | 66 |
| | | 4 | 44 | 43 |
| | | 5 | 75 | 78 |
| | | 6 | 12 | 5 |
| | | 7 | 44 | 41 |
| | | 8 | 42 | 41 |
| | | 9 | 68 | 67 |
| | | 11 | 22 | 26 |
| | | 12 | 19 | 21 |
| | | 13 | 33 | 38 |
| | | 15 | 9 | 8 |
| 8 | 6 | 0 | 38 | 36 |
| | | 1 | 46 | 47 |
| | | 2 | 17 | 15 |
| | | 3 | 38 | 38 |
| | | 4 | 31 | 27 |
| | | 5 | 73 | 77 |
| | | 6 | 29 | 26 |
| | | 7 | 42 | 41 |
| | | 8 | 11 | 11 |
| | | 9 | 46 | 46 |
| | | 10 | 18 | 16 |
| | | 11 | 33 | 36 |
| | | 13 | 31 | 32 |
| | | 14 | 16 | 21 |
| | | 15 | 20 | 22 |
| 10 | 6 | 0 | 59 | 59 |
| | | 1 | 30 | 31 |
| | | 2 | 39 | 43 |
| | | 3 | 73 | 76 |
| | | 4 | 32 | 29 |
| | | 5 | 18 | 20 |
| | | 6 | 22 | 21 |
| | | 7 | 58 | 58 |
| | | 8 | 7 | 6 |
| | | 9 | 7 | 3 |
| | | 10 | 12 | 11 |
| | | 11 | 20 | 19 |
| | | 12 | 8 | 8 |
| | | 13 | 9 | 5 |
| 12 | 6 | -13 | 31 | 31 |
| | | -12 | 12 | 7 |
| | | -11 | 9 | 2 |
| | | -10 | 24 | 18 |
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| | | -6 | 15 | 11 |
| | | -5 | 41 | 40 |
| | | -4 | 52 | 57 |
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| | | -1 | 53 | 51 |
| | | 0 | 23 | 25 |
| | | 1 | 64 | 62 |
| | | 2 | 34 | 31 |
| | | 3 | 22 | 19 |
| | | 4 | 20 | 16 |
| | | 5 | 32 | 38 |
| | | 6 | 18 | 11 |
| | | 7 | 9 | 16 |
| | | 8 | 20 | 24 |
| | | 9 | 33 | 32 |
| 14 | 6 | -9 | 10 | 9 |
| | | -8 | 15 | 13 |
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| | | 4 | 8 | 6 |
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| | | 6 | 11 | 5 |
| | | 7 | 12 | 7 |
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| | | -16 | 25 | 25 |
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| | | -13 | 20 | 18 |
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| | | -5 | 46 | 47 |
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| | | -2 | 95 | 94 |
| | | -1 | 55 | 56 |
| | | 0 | 91 | 97 |
| | | 1 | 96 | 98 |
| | | 2 | 79 | 76 |
| | | 3 | 71 | 73 |
| | | 4 | 33 | 28 |
| | | 5 | 8 | 8 |
| | | 6 | 67 | 68 |
| | | 7 | 24 | 22 |
| | | 8 | 44 | 42 |
| | | 9 | 80 | 76 |
| | | 10 | 93 | 96 |
| | | 11 | 22 | 18 |
| | | 12 | 15 | 15 |
| | | 13 | 36 | 35 |
| | | 14 | 40 | 36 |
| | | 16 | 11 | 5 |
| | | 17 | 19 | 19 |
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| 3 | 7 | -18 | 9 | 11 |
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| | | -6 | 88 | 88 |
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| | | 0 | 53 | 49 |
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| | | 2 | 115 | 113 |
| | | 3 | 95 | 93 |
| | | 4 | 19 | 21 |
| | | 5 | 114 | 115 |
| | | 6 | 105 | 100 |
| | | 7 | 75 | 75 |
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| | | 8 | 7 | 3 |
| | | 9 | 10 | 9 |
| | | 10 | 64 | 62 |
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| | | 4 | 31 | 32 |
| | | 5 | 58 | 57 |
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| | | 3 | 18 | 15 |
| | | 4 | 17 | 15 |
| | | 5 | 46 | 49 |
| | | 6 | 18 | 19 |
| | | 7 | 13 | 14 |
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| | | 2 | 46 | 47 |
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| | | 9 | 17 | 19 |
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| | | 11 | 12 | 15 |
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| 5 | 13 | -12 | 7 | 8 |
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| | | 2 | 12 | 10 |
| | | 3 | 22 | 25 |
| | | 4 | 43 | 46 |
| | | 5 | 14 | 13 |
| | | 6 | 7 | 10 |
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| | | 8 | 33 | 29 |
| | | 9 | 10 | 9 |
| | | 10 | 15 | 15 |
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| | | -5 | 25 | 23 |
| | | -4 | 46 | 49 |
| | | -3 | 14 | 13 |
| | | -2 | 22 | 20 |
| | | 0 | 44 | 44 |
| | | 1 | 14 | 13 |
| | | 2 | 22 | 22 |
| | | 3 | 19 | 17 |
| | | 4 | 47 | 44 |
| | | 5 | 13 | 9 |
| | | 6 | 16 | 15 |
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| | | 2 | 30 | 29 |
| | | 3 | 6 | 3 |
| | | 4 | 8 | 1 |
| | | 5 | 12 | 6 |
| | | 6 | 28 | 29 |
| 0 | 14 | 0 | 13 | 9 |
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| | | 2 | 19 | 15 |
| | | 3 | 37 | 39 |
| | | 5 | 26 | 27 |
| | | 6 | 17 | 14 |
| | | 7 | 13 | 10 |
| | | 9 | 37 | 37 |
| | | 10 | 13 | 10 |
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| | | -9 | 6 | 4 |
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| | | -2 | 8 | 5 |
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| | | 4 | 7 | 4 |
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| | | 6 | 33 | 36 |
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| | | 8 | 7 | 1 |
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| | | 2 | 18 | 16 |
| | | 3 | 8 | 1 |
| 0 | 16 | 0 | 14 | 12 |
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| | | 3 | 24 | 22 |
| | | 4 | 11 | 10 |
| | | 5 | 22 | 21 |
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| 4 | 16 | -5 | 22 | 26 |
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| | | -3 | 17 | 20 |
| | | -2 | 16 | 14 |
| | | -1 | 29 | 32 |
| | | 0 | 8 | 10 |
| | | 1 | 11 | 8 |
| | | 2 | 19 | 19 |
| | | 3 | 22 | 19 |

Moore (1963) was used. The anomalous part of the scattering factor of $Br^-$ was taken from Rietveld (1966). The refinement was considered to be completed when all shifts were far below the estimated standard deviations; the resulting $R$ was 0·064. The final positional and thermal parameters are presented in Table 2. Observed and calculated structure factors are given in Table 3.

## Discussion of the structure

The molecular dimensions are given in Fig. 1. The molecule, which is in the all-*trans* configuration, is nearly planar, with a maximum distance to the least-squares plane of 0·14 Å; the torsion angles are relatively small (Fig. 1). It appears that there are, within the limit of error, no alternating C–C bond distances in the pentadienylidene chain. This cannot be ascribed to disorder; in that case we would expect non-planar bond configurations around the nitrogen atoms. Consequently we have here a linear bonding system of an aromatic character with an equal contribution of two mesomeric forms which are equivalent by symmetry. The same situation was found in dipyrrolidyl-trimethincyanin perchlorate ethanol (Zedler & Kulpe, 1970).

Table 4. *Environment of the water molecule*

| Distance | Symmetry transformation | | Angle | | |
|---|---|---|---|---|---|
| ···Br(1) | $x, \quad y, \quad z$ | 3·364 Å | | Br(1)–O–Br(1′) | 89·3° |
| ···Br(1′) | $-x, \quad y, \tfrac{1}{2}-z$ | | | C(5)–O–C(5′) | 93·7 |
| ···C(5) | $-\tfrac{1}{2}+x, \tfrac{1}{2}+y, \quad z$ | 3·367 | Br(1)–O–C(5) | Br(1′)–O–C(5′) | 128·1 |
| ···C(5′) | $\tfrac{1}{2}-x, \tfrac{1}{2}+y, \tfrac{1}{2}-z$ | | Br(1)–O–C(5′) | Br(1′)–O–C(5) | 111·8 |

Table 5. *Geometry of the hydrogen bonds*

| *D* | H | *A* | Symmetry transformation of *A* | *D*–H | H···*A* | *D*···*A* | *D*–H···*A* |
|---|---|---|---|---|---|---|---|
| O | H(1) | Br | $x, \quad y, \quad z$ | 0·86 Å | 2·56 Å | 3·364 Å | 155·6° |
| N(1) | H(2) | Br | $\tfrac{1}{2}-x, \quad \tfrac{1}{2}-y, \ -z$ | 0·99 | 2·43 | 3·387 | 159·5 |
| N(2) | H(3) | Br | $x, \quad y, \quad z$ | 0·94 | 2·46 | 3·387 | 166·1 |
| C(5) | H(8) | O | $\tfrac{1}{2}+x, \ -\tfrac{1}{2}+y, \quad z$ | 0·98 | 2·44 | 3·367 | 156·8 |

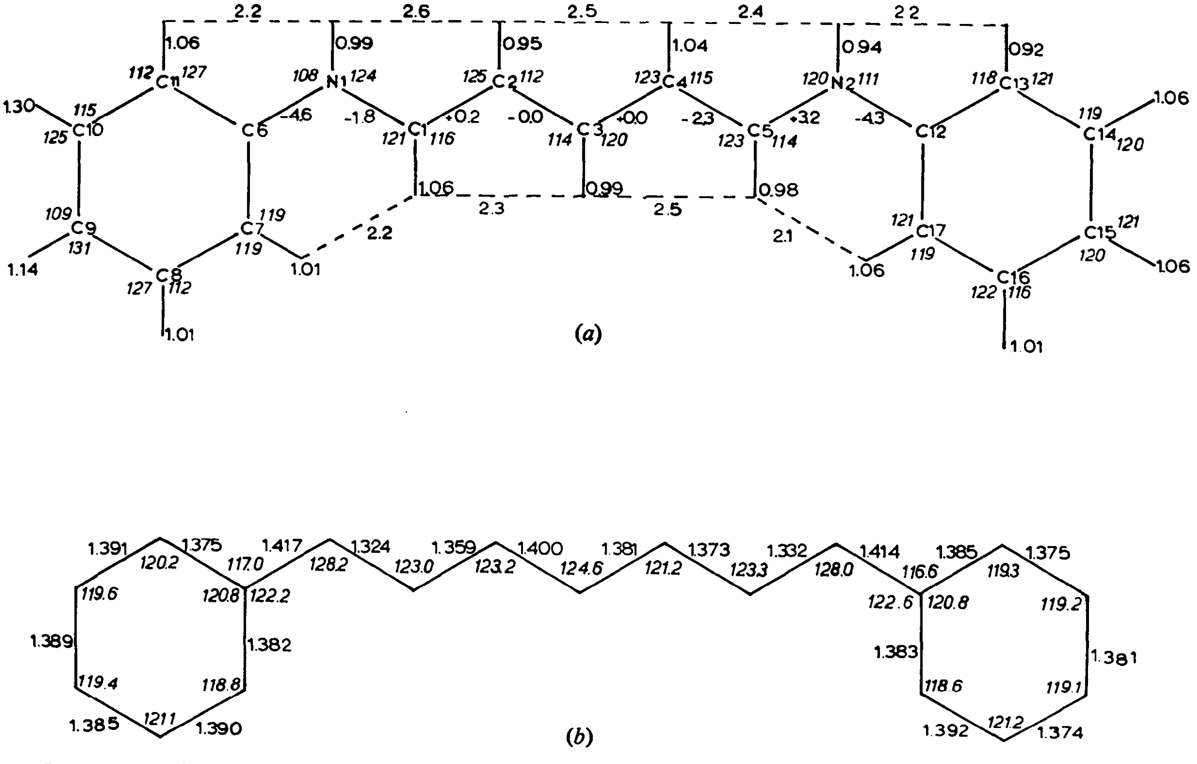

Fig. 1. Interatomic distances and bond angles. (*a*) Torsion angles in the chain calculated from the heavy-atom positions are given by numbers provided with a sign. The estimated standard deviations of the other values that give bond lengths, bond angles and non-bonded distances in which hydrogen atoms are involved are 0·08 Å, 4° and 0·1 Å respectively. (*b*) Bond lengths and bond angles involving the heavy atoms. The e.s.d.'s are 0·008 Å and 0·5° respectively.

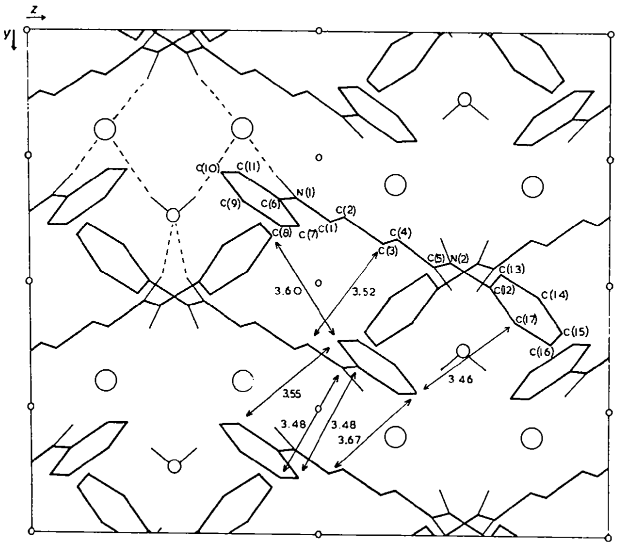

Fig. 2. The structure of *N*-phenyl-(5-anilino-2,4-pentadienylidene)ammonium bromide hemihydrate projected along the *a* axis. Some of the hydrogen bonds are depicted; the shortest intermolecular contacts are indicated.

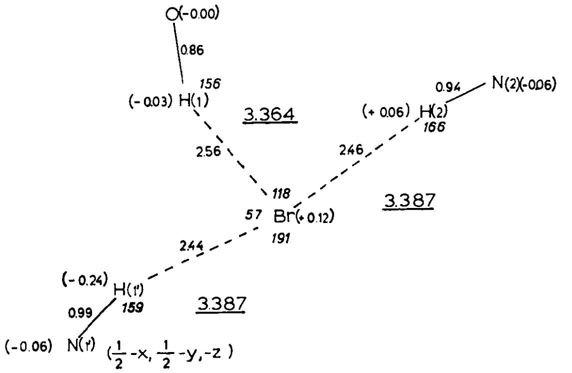

Fig. 3. The environment of the bromine ion. The underlined values are related to the distances between the bromine ion and the O and N atoms. The values in parentheses give the distances of the atoms to the mean plane through Br, N(1'), N(2) and O.

It can also be expected in the structure of 3,3′-diethylthiacarbocyanine bromide (Wheatley, 1959); here, however, the accuracy of the X-ray results did not permit conclusions about the bond orders in the methyne chain to be drawn.

The fact that the angle C(7)C(6)N(1) is significantly larger than the angle C(11)C(6)N(1) can be explained by the repulsion between H(4) and H(9). The same holds for the angle C(17)C(12)N(2) with respect to C(13)C(12)N(2), which will be influenced by the small contact between H(8) and H(18). A similar effect is found in the centric molecule diphenyl-1,3,5,7-octatetraene (Drenth & Wiebenga, 1955). The values of the pertinent angles are 122·7 and 119·2° respectively.

The water molecule (O–H = 0·86 Å; ∠H–O–H = 102°), which is situated on the twofold axis, plays a role as proton donor for two bromine ions, and has two close contacts involving hydrogen atoms from symmetry-related carbon atoms which are bonded to the nitrogen atoms in the pentadienylidene chain (Fig. 2). Thus the water molecule participates in four bonds of the type X–H···Y in a pseudo-tetrahedral arrangement (Table 4).

Another hydrogen-bonding system consists of two N–H···$Br^-$ contacts (Table 5), which, with a third hydrogen bond to a water molecule, form a nearly planar asymmetric arrangement (Fig. 3).

The remaining intermolecular contacts, mainly involving one phenyl group, can be inferred from Fig. 2.

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