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# Barium Aluminate Hydrates. III. The Crystal Structure of $\alpha$-$Ba[AlO(OH)_2]_2$

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The compound $\alpha$-$BaO.Al_2O_3.2H_2O$ has been shown by X-ray structure analysis to contain complex anions of formula $[AlO(OH)_2^-]_\infty$. The structure of this compound is essentially an arrangement of single helical chains formed from $AlO_2(OH)_2$ tetrahedra linked by sharing corners. The chains are held together by barium atoms and H-bonds. The formula should thus be written $\alpha$-$Ba[AlO(OH)_2]_2$.

## Introduction

The compound of empirical formula $BaO.Al_2O_3.2H_2O$ was first prepared by Carlson & Wells (1948) by treating $BaO.Al_2O_3.7H_2O$ hydrothermally at 124–215°C under saturated steam pressure. Later its stability relationships were studied (Carlson, Chaconas & Wells, 1950). Several polymorphs have been reported (Thilo & Gessner, 1965; Ahmed, 1969). The one discussed in this paper is the same as that prepared by Carlson *et al.* and will be called $\alpha$-$BaO.Al_2O_3.2H_2O$.

In the present study pure $\alpha$-$BaO.Al_2O_3.2H_2O$ was prepared hydrothermally using different starting materials having bulk molar $BaO/Al_2O_3$ ratio 1·0 at 80–215°C under saturated water vapour pressure. The refractive indices of this compound (Table 1) agreed well with those reported by Carlson & Wells (1948).

Table 1. *Crystal data for* $\alpha$-$Ba[AlO(OH)_2]_2$

| Unit-cell | |
|---|---|
| $a$ | 12·99 Å |
| $b$ | 12·34 |
| $c$ | 7·19 |
| Space group *Pbca* | |
| $Z$ | 8 |
| X-ray density | 3·356 g.cm$^{-3}$ |
| Observed density | 3·30 g.cm$^{-3}$ |
| $\mu$ | 72 cm$^{-1}$ |

Refractive indices

| | Carlson & Wells (1948) | Present study |
|---|---|---|
| $\alpha$ | 1·610 | 1·607 ± 0·003 |
| $\gamma$ | 1·613 | 1·613 ± 0·003 |

Single-crystal X-ray studies confirmed that the crystals were orthorhombic. Unit-cell constants and space group are given in Table 1. The cell contents are $Ba_8Al_{16}O_{48}H_{32}$.

## Experimental

The crystal selected for intensity measurements was a lath whose longest dimension was less than 0·2 mm. Three-dimensional intensity data for about 3600 reflexions in half of the reciprocal lattice were collected using a Hilger and Watts automatic linear diffractometer with Mo $K\alpha$ radiation. Each reflexion was measured four times and no correction was made for absorption or extinction. Data were processed on an Elliott 803B computer, mainly using programs kindly supplied by Daly, Stephens & Wheatley (1963). After averaging equivalent reflexions, values for 943 independent reflexions remained.

## Structure determination

The approximate positions of the barium atoms were derived from the Patterson function, and a three-dimensional Fourier synthesis, based on the signs obtained using the positional parameters of the barium atoms, indicated plausible sites for the aluminum and oxygen atoms. The structure was refined by the method of least squares (block-diagonal approximation). Scattering factors for $Ba^{2+}$ and $Al^{3+}$ were taken from *International Tables for X-ray Crystallography* (1962). A curve for $O^{2-}$ was constructed by extrapolation from values for $O^-$. It was hoped that this would to some extent compensate for ignoring the contribution of the hydrogen atoms.

The least-squares procedure closely followed that of Cruickshank, Pilling, Bujosa, Lovell & Truter (1961) and incorporated the same weighting scheme and method of calculation of the estimated standard deviations. At first positional parameters only were refined, using low angle data only (116 reflexions); after four further cycles on the full data (943 reflexions) with isotropic temperature factors the maximum shift in atomic coordinates was about $\frac{1}{3}$ of the estimated standard deviation (e.s.d.) and the average shift only $\frac{1}{8}$ of the average e.s.d., showing that the structure was refined as completely as the data would permit. The conventional $R$ value at this point was 0·09. Table 2 gives the values of $F_o$ and $F_c$ obtained, Table 3 the corresponding final parameters, and Table 4 some details of the coordination polyhedra. A three-dimensional difference map gave electron density values ranging from about $-1$ e.Å$^{-3}$ to $+1$ e.Å$^{-3}$, except in

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Table 2. *Observed and calculated structure factors* ( × 10)

| h | k | $F_o$ | $F_c$ |
|---|---|---|---|
| L= 0 | | | |
| 2 | 0 | 1030 | 1501 |
| 4 | 0 | -1540 | -2248 |
| 6 | 0 | -2095 | -3175 |
| 8 | 0 | -1698 | -2589 |
| 10 | 0 | -439 | -556 |
| 2 | 1 | -1487 | -1804 |
| 4 | 1 | -2147 | -3105 |
| 6 | 1 | -809 | -1088 |
| 8 | 1 | 1109 | 1615 |
| 10 | 1 | 898 | 1397 |
| 0 | 2 | -1108 | -915 |
| 2 | 2 | -1056 | -991 |
| 4 | 2 | -589 | -598 |
| 6 | 2 | 1127 | 1428 |
| 8 | 2 | 1092 | 1475 |
| 2 | 3 | -413 | -377 |
| 4 | 3 | -1795 | -1861 |
| 6 | 3 | -140 | -28 |
| 10 | 3 | 731 | 963 |
| 0 | 4 | -1954 | -1915 |
| 2 | 4 | -1936 | -1707 |
| 4 | 4 | 757 | 784 |
| 6 | 4 | 1215 | 1409 |
| 8 | 4 | 1470 | 1848 |
| 2 | 5 | 2430 | 2181 |
| 4 | 5 | 3848 | 4066 |
| 6 | 5 | 1030 | 1099 |
| 8 | 5 | -1654 | -1915 |
| 10 | 5 | -1390 | -1710 |
| 0 | 6 | 3249 | 3349 |
| 2 | 6 | 1840 | 1677 |
| 4 | 6 | -1549 | -1512 |
| 6 | 6 | -1320 | -1361 |
| 8 | 6 | -1795 | -1973 |
| 10 | 6 | -378 | -373 |
| 2 | 7 | -1461 | -1369 |
| 4 | 7 | -1566 | -1460 |
| 6 | 7 | 203 | 192 |
| 10 | 7 | 766 | 857 |
| 0 | 8 | 1250 | 1143 |
| 4 | 8 | -686 | -675 |
| 2 | 9 | -1258 | -1139 |
| 4 | 9 | -1249 | -1223 |
| 8 | 9 | 493 | 469 |
| 10 | 9 | 405 | 447 |
| 0 | 10 | -2033 | -2023 |
| 2 | 10 | -580 | -600 |
| 6 | 10 | 1374 | 1312 |
| 8 | 10 | 1646 | 1643 |
| 2 | 11 | 1065 | 1015 |
| 4 | 11 | 2122 | 2122 |
| 6 | 11 | 560 | 616 |
| 8 | 11 | -1161 | -1161 |
| 10 | 11 | -968 | -932 |
| 0 | 12 | 1514 | 1472 |
| 2 | 12 | 616 | 568 |
| 4 | 12 | -483 | -485 |
| 6 | 12 | -756 | -761 |
| 8 | 12 | -756 | -743 |
| 2 | 13 | -510 | -548 |
| 6 | 13 | -254 | -235 |
| 0 | 14 | 792 | 717 |
| 2 | 14 | 317 | 259 |
| 4 | 14 | -272 | -256 |
| L= 1 | | | |
| 1 | 1 | 951 | 825 |
| 2 | 1 | 792 | 697 |
| 3 | 1 | 1065 | 969 |
| 4 | 1 | 643 | 622 |
| 5 | 1 | -589 | -595 |
| 6 | 1 | 1127 | 1066 |
| 7 | 1 | -2024 | -1999 |
| 8 | 1 | -554 | -535 |
| 10 | 1 | -888 | -880 |
| 11 | 1 | 537 | 506 |
| 12 | 1 | -263 | -240 |
| 13 | 1 | 502 | 478 |
| 0 | 2 | 1004 | 850 |
| 1 | 2 | -1381 | -1108 |
| 2 | 2 | 2254 | 1916 |
| 3 | 2 | -2667 | -2441 |
| 4 | 2 | -422 | -369 |
| 5 | 2 | -1117 | -1121 |
| 6 | 2 | -2147 | -2076 |
| 8 | 2 | -879 | -848 |
| 9 | 2 | 1189 | 1126 |
| 11 | 2 | 1206 | 1155 |
| 12 | 2 | 493 | 451 |
| 13 | 2 | 405 | 376 |
| 14 | 2 | 1021 | 953 |
| 1 | 3 | -2711 | -2292 |
| 2 | 3 | -3671 | -3227 |
| 3 | 3 | -457 | -383 |
| 4 | 3 | -1901 | -1650 |
| 5 | 3 | 1567 | 1413 |
| 6 | 3 | -378 | -369 |
| 7 | 3 | 1655 | 1552 |
| 8 | 3 | 282 | 279 |
| 9 | 3 | 1312 | 1245 |
| 10 | 3 | 1937 | 1860 |
| 11 | 3 | -272 | -224 |
| 12 | 3 | 713 | 658 |
| 13 | 3 | -1390 | -1314 |
| 14 | 3 | -422 | -376 |
| 0 | 4 | -1496 | -1237 |
| 1 | 4 | 1532 | 1265 |
| 2 | 4 | -844 | -721 |
| 3 | 4 | 1708 | 1488 |
| 4 | 4 | 194 | 176 |
| 5 | 4 | 1479 | 1294 |
| 6 | 4 | 1418 | 1246 |
| 8 | 4 | 519 | 490 |
| 9 | 4 | -1135 | -1044 |
| 10 | 4 | 229 | 177 |
| 11 | 4 | -1126 | -1041 |
| 12 | 4 | -307 | -285 |
| 13 | 4 | -272 | -221 |
| 14 | 4 | -879 | -802 |
| 1 | 5 | 1374 | 1056 |
| 2 | 5 | -272 | -196 |
| 3 | 5 | 317 | 238 |
| 4 | 5 | 775 | 663 |
| 5 | 5 | -1003 | -851 |
| 6 | 5 | 564 | 463 |
| 7 | 5 | -1003 | -877 |
| 8 | 5 | -272 | -251 |
| 10 | 5 | -430 | -376 |
| 11 | 5 | 291 | 291 |
| 0 | 6 | -1126 | -989 |
| 1 | 6 | 1065 | 912 |
| 2 | 6 | -413 | -389 |
| 3 | 6 | 845 | 773 |
| 5 | 6 | 185 | 185 |
| 6 | 6 | 986 | 868 |
| 8 | 6 | 343 | 248 |
| 9 | 6 | -210 | -140 |
| 10 | 6 | 229 | 210 |
| 11 | 6 | -518 | -480 |
| 13 | 6 | -254 | -265 |
| 14 | 6 | -721 | -631 |
| 1 | 7 | 1444 | 1247 |
| 2 | 7 | 969 | 855 |
| 3 | 7 | 757 | 699 |
| 4 | 7 | 696 | 610 |
| 5 | 7 | -1390 | -1178 |
| 6 | 7 | 784 | 686 |
| 7 | 7 | -1584 | -1434 |
| 8 | 7 | -325 | -264 |
| 9 | 7 | -483 | -451 |
| 10 | 7 | -1249 | -1113 |
| 11 | 7 | 572 | 502 |
| 12 | 7 | -237 | -183 |
| 13 | 7 | 634 | 548 |
| 0 | 8 | 1294 | 1134 |
| 1 | 8 | -545 | -510 |
| 2 | 8 | 1620 | 1437 |
| 3 | 8 | -2033 | -1861 |
| 4 | 8 | -193 | -149 |
| 5 | 8 | -1936 | -1688 |
| 6 | 8 | -2227 | -1976 |
| 8 | 8 | -1003 | -853 |
| 9 | 8 | 1699 | 1510 |
| 11 | 8 | 1294 | 1135 |
| 12 | 8 | 511 | 423 |
| 1 | 9 | -1839 | -1631 |
| 2 | 9 | -1601 | -1441 |
| 4 | 9 | -615 | -548 |
| 5 | 9 | 1092 | 968 |
| 6 | 9 | -457 | -376 |
| 7 | 9 | 1197 | 1015 |
| 8 | 9 | 440 | 391 |
| 9 | 9 | 660 | 552 |
| 10 | 9 | 933 | 792 |
| 12 | 9 | 564 | 437 |
| 0 | 10 | -950 | -888 |
| 1 | 10 | 660 | 574 |
| 2 | 10 | -298 | -260 |
| 3 | 10 | 1171 | 1005 |
| 4 | 10 | 247 | 259 |
| 5 | 10 | 405 | 341 |
| 6 | 10 | 396 | 335 |
| 8 | 10 | 414 | 357 |
| 9 | 10 | -457 | -425 |
| 10 | 10 | 291 | 249 |
| 11 | 10 | -730 | -618 |
| 1 | 11 | -325 | -326 |
| 2 | 11 | -615 | -643 |
| 3 | 11 | 229 | 163 |
| 10 | 11 | 379 | 358 |
| 0 | 12 | -351 | -317 |
| 1 | 12 | 528 | 541 |
| 2 | 12 | -545 | -589 |
| 3 | 12 | 880 | 921 |
| 4 | 12 | 299 | 278 |
| 5 | 12 | 396 | 437 |
| 6 | 12 | 511 | 536 |
| L= 2 | | | |
| 2 | 0 | 1215 | 1204 |
| 3 | 0 | 3152 | 3483 |
| 4 | 0 | -686 | -680 |
| 5 | 0 | 2025 | 2082 |
| 6 | 0 | -985 | -1024 |
| 9 | 0 | -1434 | -1447 |
| 10 | 0 | -184 | -167 |
| 11 | 0 | -2112 | -2132 |
| 13 | 0 | -862 | -827 |
| 14 | 0 | 696 | 689 |
| 2 | 1 | -1126 | -1162 |
| 3 | 1 | 414 | 408 |
| 4 | 1 | -809 | -806 |
| 5 | 1 | -1619 | -1760 |
| 6 | 1 | -457 | -472 |
| 7 | 1 | -1434 | -1620 |
| 9 | 1 | -1188 | -1211 |
| 10 | 1 | 1145 | 1164 |
| 11 | 1 | 722 | 710 |
| 13 | 1 | 1048 | 1061 |
| 0 | 2 | 537 | 464 |
| 1 | 2 | 379 | 326 |
| 2 | 2 | -563 | -540 |
| 3 | 2 | -2323 | -2314 |
| 5 | 2 | -1329 | -1423 |
| 6 | 2 | 396 | 383 |
| 7 | 2 | 308 | 331 |
| 9 | 2 | 1303 | 1346 |
| 11 | 2 | 528 | 490 |
| 1 | 3 | 1171 | 1118 |
| 2 | 3 | -140 | -166 |
| 3 | 3 | -149 | -103 |
| 5 | 3 | -607 | -589 |
| 6 | 3 | -457 | -446 |
| 7 | 3 | -439 | -449 |
| 8 | 3 | 220 | 223 |
| 9 | 3 | -360 | -392 |
| 10 | 3 | 291 | 311 |
| 13 | 3 | 414 | 398 |
| 14 | 3 | -254 | -230 |
| 0 | 4 | -1346 | -1269 |
| 1 | 4 | -607 | -597 |
| 2 | 4 | -800 | -781 |
| 3 | 4 | -1725 | -1834 |
| 4 | 4 | 264 | 292 |
| 5 | 4 | -2033 | -2126 |
| 6 | 4 | 1374 | 1393 |
| 7 | 4 | 599 | 589 |
| 9 | 4 | 1558 | 1603 |
| 11 | 4 | 704 | 722 |
| 14 | 4 | -563 | -551 |
| 1 | 5 | -2500 | -2753 |
| 2 | 5 | 1101 | 1094 |
| 3 | 5 | -386 | -389 |
| 4 | 5 | 176 | 71 |
| 5 | 5 | 1241 | 1271 |
| 6 | 5 | 502 | 517 |
| 7 | 5 | 2774 | 2829 |
| 8 | 5 | -193 | -158 |
| 9 | 5 | 845 | 858 |
| 10 | 5 | -765 | -790 |
| 11 | 5 | -554 | -547 |
| 13 | 5 | -1364 | -1324 |
| 14 | 5 | 255 | 207 |
| 1 | 6 | 1294 | 1345 |
| 2 | 6 | 458 | 457 |
| 3 | 6 | 2096 | 2208 |
| 4 | 6 | 335 | 308 |
| 5 | 6 | 1206 | 1238 |
| 6 | 6 | -879 | -850 |
| 8 | 6 | -290 | -233 |
| 9 | 6 | -968 | -949 |
| 11 | 6 | -1505 | -1505 |
| 13 | 6 | -668 | -613 |
| 14 | 6 | 493 | 513 |
| 1 | 7 | 1532 | 1609 |
| 2 | 7 | -598 | -588 |
| 4 | 7 | 194 | 133 |
| 5 | 7 | -906 | -916 |
| 7 | 7 | -976 | -959 |
| 9 | 7 | -756 | -705 |
| 11 | 7 | 247 | 256 |
| 13 | 7 | 995 | 841 |
| 1 | 8 | -219 | -189 |
| 2 | 8 | 229 | 226 |
| 3 | 8 | 220 | 203 |
| 5 | 8 | -501 | -487 |
| 7 | 8 | 238 | 233 |
| 9 | 8 | 291 | 310 |
| 11 | 8 | -237 | -211 |
| 1 | 9 | 1215 | 1314 |
| 2 | 9 | -695 | -717 |
| 5 | 9 | -783 | -780 |
| 7 | 9 | -563 | -580 |
| 9 | 9 | -695 | -695 |
| 10 | 9 | 484 | 483 |
| 11 | 9 | 299 | 284 |
| 0 | 10 | 238 | 202 |
| 1 | 10 | -615 | -615 |
| 2 | 10 | -571 | -604 |
| 3 | 10 | -1575 | -1685 |
| 5 | 10 | -1417 | -1490 |
| 6 | 10 | 704 | 727 |
| 7 | 10 | 370 | 378 |
| 8 | 10 | 255 | 170 |
| 9 | 10 | 1250 | 1224 |
| 1 | 11 | -1240 | -1322 |
| 2 | 11 | 431 | 468 |
| 3 | 11 | -580 | -623 |
| 5 | 11 | 969 | 1009 |
| 7 | 11 | 1541 | 1559 |
| 9 | 11 | 775 | 763 |
| 1 | 12 | 335 | 374 |
| 2 | 12 | 229 | 220 |
| 3 | 12 | 1039 | 1117 |
| 5 | 12 | 502 | 535 |
| 1 | 13 | 379 | 562 |
| 1 | 15 | 652 | 983 |
| 1 | 16 | -307 | -422 |
| L= 3 | | | |
| 2 | 1 | 564 | 604 |
| 3 | 1 | -219 | -236 |
| 4 | 1 | 1752 | 1867 |
| 5 | 1 | 933 | 970 |
| 6 | 1 | 792 | 795 |
| 7 | 1 | 1162 | 1172 |
| 8 | 1 | -1214 | -1223 |
| 9 | 1 | -228 | -225 |
| 10 | 1 | -642 | -641 |
| 11 | 1 | -219 | -210 |
| 12 | 1 | -474 | -462 |
| 13 | 1 | -325 | -305 |
| 0 | 2 | 2729 | 2923 |
| 1 | 2 | 440 | 443 |
| 2 | 2 | 1426 | 1448 |
| 3 | 2 | 1153 | 1147 |
| 4 | 2 | -756 | -745 |
| 5 | 2 | 898 | 888 |
| 6 | 2 | -1549 | -1610 |
| 7 | 2 | 203 | 197 |
| 8 | 2 | -1971 | -2049 |
| 9 | 2 | -1012 | -1009 |
| 11 | 2 | -791 | -778 |
| 12 | 2 | 1303 | 1260 |
| 14 | 2 | 854 | 787 |
| 1 | 3 | 1286 | 1305 |
| 2 | 3 | -2147 | -2225 |
| 3 | 3 | 273 | 244 |
| 4 | 3 | -2605 | -2713 |
| 5 | 3 | -915 | -939 |
| 6 | 3 | -334 | -330 |
| 7 | 3 | -739 | -719 |
| 8 | 3 | 863 | 865 |
| 9 | 3 | -598 | -566 |
| 10 | 3 | 1356 | 1321 |
| 12 | 3 | 1374 | 1330 |
| 13 | 3 | 766 | 740 |
| 0 | 4 | -2711 | -2775 |
| 1 | 4 | -985 | -945 |
| 2 | 4 | -703 | -713 |
| 3 | 4 | -545 | -525 |
| 4 | 4 | 643 | 626 |
| 5 | 4 | -237 | -252 |
| 6 | 4 | 1206 | 1175 |
| 8 | 4 | 1312 | 1292 |
| 9 | 4 | 326 | 318 |
| 10 | 4 | 273 | 250 |
| 11 | 4 | 387 | 392 |
| 12 | 4 | -853 | -832 |
| 14 | 4 | -835 | -805 |
| 2 | 5 | -157 | -98 |
| 3 | 5 | -448 | -442 |
| 4 | 5 | 748 | 700 |
| 5 | 5 | 158 | 148 |
| 6 | 5 | 511 | 460 |
| 7 | 5 | 704 | 668 |
| 8 | 5 | -518 | -505 |
| 10 | 5 | -413 | -389 |
| 11 | 5 | -281 | -305 |
| 0 | 6 | -1373 | -1452 |
| 1 | 6 | -659 | -640 |
| 3 | 6 | -774 | -705 |
| 4 | 6 | 361 | 346 |
| 5 | 6 | -474 | -433 |
| 6 | 6 | 291 | 299 |
| 8 | 6 | 643 | 650 |
| 9 | 6 | 528 | 498 |
| 10 | 6 | 247 | 208 |
| 11 | 6 | 537 | 491 |
| 12 | 6 | -492 | -471 |
| 1 | 7 | -536 | -529 |
| 2 | 7 | 696 | 688 |
| 3 | 7 | -580 | -572 |
| 4 | 7 | 2184 | 2250 |
| 5 | 7 | 854 | 835 |
| 6 | 7 | 590 | 588 |
| 7 | 7 | 801 | 762 |
| 8 | 7 | -1091 | -1057 |
| 10 | 7 | -1047 | -967 |
| 11 | 7 | -386 | -360 |
| 12 | 7 | -615 | -594 |
| 13 | 7 | -351 | -236 |
| 0 | 8 | 2078 | 2152 |
| 1 | 8 | 423 | 430 |
| 2 | 8 | 1109 | 1125 |
| 3 | 8 | 1057 | 1015 |
| 4 | 8 | -448 | -525 |
| 5 | 8 | 537 | 524 |
| 6 | 8 | -1434 | -1420 |
| 8 | 8 | -1663 | -1604 |
| 9 | 8 | -651 | -618 |
| 11 | 8 | -642 | -555 |
| 12 | 8 | 995 | 973 |
| 1 | 9 | 203 | 182 |
| 2 | 9 | -1267 | -1299 |
| 3 | 9 | 211 | 161 |
| 4 | 9 | -1505 | -1492 |
| 6 | 9 | -466 | -416 |
| 7 | 9 | -298 | -310 |
| 8 | 9 | 704 | 659 |
| 9 | 9 | -254 | -240 |
| 10 | 9 | 942 | 879 |
| 0 | 10 | -941 | -942 |
| 1 | 10 | -290 | -248 |
| 2 | 10 | -342 | -361 |
| 3 | 10 | -439 | -492 |
| 4 | 10 | 387 | 413 |
| 6 | 10 | 379 | 361 |
| 8 | 10 | 458 | 381 |
| 10 | 10 | 264 | 227 |
| 4 | 11 | -237 | -261 |
| 7 | 11 | -325 | -269 |
| 0 | 12 | -1355 | -1478 |
| 1 | 12 | -457 | -486 |
| 2 | 12 | -386 | -380 |
| 3 | 12 | -386 | -439 |
| 4 | 12 | 546 | 573 |
| 6 | 12 | 387 | 369 |
| 1 | 13 | -316 | -416 |
| 2 | 13 | 484 | 663 |
| 4 | 13 | 969 | 1374 |
| 5 | 13 | 291 | 404 |
| 0 | 14 | 766 | 1068 |
| L= 4 | | | |
| 2 | 0 | -1196 | -1304 |
| 3 | 0 | 1021 | 1029 |
| 4 | 0 | 502 | 483 |
| 5 | 0 | 652 | 624 |
| 6 | 0 | 2148 | 2114 |
| 8 | 0 | 1233 | 1181 |
| 9 | 0 | -871 | -840 |
| 10 | 0 | 405 | 407 |
| 11 | 0 | -818 | -773 |
| 12 | 0 | -644 | -797 |
| 14 | 0 | -1557 | -1431 |
| 2 | 1 | 1611 | 1904 |
| 3 | 1 | 308 | 331 |
| 4 | 1 | 1145 | 1230 |
| 5 | 1 | -791 | -834 |
| 6 | 1 | 511 | 510 |
| 7 | 1 | -1188 | -1204 |
| 8 | 1 | -413 | -400 |
| 9 | 1 | -202 | -181 |
| 10 | 1 | -1346 | -1349 |
| 12 | 1 | -651 | -620 |
| 13 | 1 | 687 | 671 |
| 0 | 2 | 132 | 93 |
| 1 | 2 | -254 | -248 |
| 2 | 2 | 951 | 972 |
| 3 | 2 | 229 | 214 |
| 4 | 2 | 150 | 146 |
| 5 | 2 | -325 | -355 |
| 6 | 2 | -1082 | -1108 |
| 8 | 2 | -695 | -693 |
| 9 | 2 | 220 | 204 |
| 10 | 2 | 203 | 171 |
| 12 | 2 | 238 | 226 |
| 14 | 2 | 299 | 290 |
| 2 | 3 | 625 | 624 |
| 4 | 3 | 273 | 207 |
| 5 | 3 | -281 | -283 |
| 6 | 3 | -263 | -220 |
| 7 | 3 | 273 | 152 |
| 8 | 3 | 264 | 269 |
| 10 | 3 | -360 | -317 |
| 0 | 4 | -184 | -16 |
| 1 | 4 | -184 | -184 |
| 2 | 4 | 1145 | 1184 |
| 3 | 4 | -853 | -872 |
| 4 | 4 | 370 | 387 |
| 5 | 4 | -571 | -573 |
| 6 | 4 | -1505 | -1535 |
| 7 | 4 | -307 | -303 |
| 8 | 4 | -932 | -928 |
| 9 | 4 | 696 | 704 |
| 11 | 4 | 652 | 635 |
| 12 | 4 | 387 | 348 |
| 14 | 4 | 581 | 545 |
| 1 | 5 | -721 | -762 |
| 2 | 5 | -1734 | -1844 |
| 3 | 5 | -615 | -603 |
| 4 | 5 | -1469 | -1489 |
| 5 | 5 | 977 | 980 |
| 6 | 5 | -985 | -977 |
| 7 | 5 | 828 | 815 |
| 8 | 5 | 880 | 854 |
| 9 | 5 | 308 | 269 |
| 10 | 5 | 1541 | 1509 |
| 11 | 5 | -237 | -219 |
| 12 | 5 | 634 | 607 |
| 13 | 5 | -439 | -427 |
| 14 | 5 | -272 | -193 |
| 0 | 6 | -2006 | -2117 |
| 1 | 6 | 660 | 751 |
| 2 | 6 | -1293 | -1337 |
| 3 | 6 | 1391 | 1375 |
| 4 | 6 | 942 | 953 |
| 5 | 6 | 379 | 374 |
| 6 | 6 | 1497 | 1530 |
| 8 | 6 | 880 | 834 |
| 9 | 6 | -739 | -691 |
| 10 | 6 | 335 | 330 |
| 11 | 6 | -774 | -724 |
| 12 | 6 | -677 | -660 |
| 1 | 7 | 352 | 350 |
| 2 | 7 | 1039 | 1095 |
| 3 | 7 | 247 | 225 |
| 4 | 7 | 933 | 952 |
| 5 | 7 | -395 | -370 |
| 6 | 7 | 282 | 287 |
| 7 | 7 | -298 | -296 |
| 9 | 7 | -351 | -305 |
| 10 | 7 | -1056 | -1038 |
| 12 | 7 | -518 | -470 |
| 13 | 7 | 326 | 254 |
| 0 | 8 | 211 | 78 |
| 1 | 8 | 255 | 309 |
| 4 | 8 | 247 | 204 |
| 6 | 8 | -228 | -212 |
| 8 | 8 | -219 | -177 |
| 1 | 9 | 326 | 357 |
| 2 | 9 | 925 | 1011 |
| 4 | 9 | 370 | 336 |
| 5 | 9 | -307 | -270 |
| 6 | 9 | 264 | 239 |
| 7 | 9 | -448 | -428 |
| 10 | 9 | -739 | -725 |
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| 1 | 10 | -518 | -536 |
| 2 | 10 | 828 | 892 |
| 3 | 10 | -518 | -529 |
| 5 | 10 | -395 | -427 |
| 6 | 10 | -1417 | -1478 |
| 8 | 10 | -739 | -693 |
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| 1 | 11 | -501 | -517 |
| 2 | 11 | -959 | -1010 |
| 3 | 11 | -316 | -313 |
| 4 | 11 | -1249 | -1311 |
| 5 | 11 | 475 | 469 |
| 6 | 11 | -668 | -670 |
| 7 | 11 | 748 | 755 |
| 8 | 11 | 704 | 710 |
| 0 | 12 | -871 | -1004 |
| 1 | 12 | 370 | 411 |
| 2 | 12 | -607 | -651 |
| 3 | 12 | 449 | 499 |
| 4 | 12 | 326 | 394 |
| 5 | 12 | 449 | 345 |
| 6 | 12 | 775 | 804 |
| 2 | 13 | 291 | 478 |
| 0 | 16 | 440 | 625 |
| L= 5 | | | |
| 2 | 1 | 387 | 383 |
| 3 | 1 | -325 | -317 |
| 4 | 1 | -351 | -352 |
| 5 | 1 | 652 | 661 |
| 6 | 1 | -351 | -364 |
| 7 | 1 | 1365 | 1422 |
| 8 | 1 | 326 | 323 |
| 9 | 1 | 379 | 385 |
| 11 | 1 | -422 | -440 |
| 13 | 1 | -510 | -479 |
| 0 | 2 | -827 | -687 |
| 1 | 2 | 555 | 493 |
| 2 | 2 | -131 | -111 |
| 3 | 2 | 2404 | 2283 |
| 4 | 2 | 405 | 352 |
| 5 | 2 | 1981 | 1928 |
| 7 | 2 | -360 | -381 |
| 8 | 2 | 370 | 385 |
| 9 | 2 | -1496 | -1472 |
| 11 | 2 | -1249 | -1223 |
| 12 | 2 | -316 | -288 |
| 13 | 2 | -386 | -342 |
| 1 | 3 | 2950 | 2567 |
| 2 | 3 | 440 | 383 |
| 3 | 3 | -334 | -312 |
| 4 | 3 | 925 | 807 |
| 5 | 3 | -1513 | -1367 |
| 6 | 3 | -166 | -133 |
| 7 | 3 | -1469 | -1382 |
| 8 | 3 | -298 | -279 |
| 9 | 3 | -1258 | -1205 |
| 10 | 3 | -210 | -173 |
| 11 | 3 | 590 | 556 |
| 12 | 3 | -272 | -255 |
| 13 | 3 | 1400 | 1247 |
| 0 | 4 | 537 | 471 |
| 1 | 4 | -1012 | -823 |
| 2 | 4 | -193 | -152 |
| 3 | 4 | -1857 | -1583 |
| 5 | 4 | -1179 | -1052 |
| 7 | 4 | 291 | 260 |
| 8 | 4 | -246 | -236 |
| 9 | 4 | 819 | 757 |
| 11 | 4 | 977 | 902 |
| 13 | 4 | 519 | 446 |
| 1 | 5 | 238 | 186 |
| 2 | 5 | 185 | 119 |
| 3 | 5 | -624 | -529 |
| 4 | 5 | -140 | -122 |
| 7 | 5 | 572 | 524 |
| 11 | 5 | -246 | -239 |
| 0 | 6 | 828 | 682 |
| 1 | 6 | -536 | -426 |
| 2 | 6 | -140 | -121 |
| 3 | 6 | -747 | -665 |
| 5 | 6 | -624 | -571 |
| 7 | 6 | 203 | 168 |
| 8 | 6 | -237 | -189 |
| 9 | 6 | 387 | 370 |
| 11 | 6 | 546 | 482 |
| 1 | 7 | -1610 | -1400 |
| 2 | 7 | 317 | 335 |
| 3 | 7 | -413 | -351 |
| 4 | 7 | -457 | -406 |
| 5 | 7 | 960 | 811 |
| 7 | 7 | 1752 | 1530 |
| 8 | 7 | 247 | 169 |
| 9 | 7 | 863 | 753 |
| 11 | 7 | -730 | -612 |
| 0 | 8 | -607 | -514 |
| 1 | 8 | 511 | 410 |
| 2 | 8 | -184 | -148 |
| 3 | 8 | 1840 | 1589 |
| 5 | 8 | 1629 | 1393 |
| 6 | 8 | 255 | 199 |
| 7 | 8 | -474 | -412 |
| 8 | 8 | 572 | 488 |
| 9 | 8 | -1214 | -1017 |
| 11 | 8 | -862 | -758 |
| 1 | 9 | 1567 | 1368 |
| 2 | 9 | 264 | 253 |
| 4 | 9 | 220 | 188 |
| 5 | 9 | -747 | -623 |
| 7 | 9 | -1320 | -1096 |
| 8 | 9 | -237 | -219 |
| 9 | 9 | -800 | -655 |
| 0 | 10 | 255 | 214 |
| 1 | 10 | -430 | -406 |
| 3 | 10 | -536 | -456 |
| 1 | 11 | 299 | 305 |
| 2 | 11 | 238 | 171 |
| 4 | 11 | 264 | 215 |
| 0 | 12 | 299 | 172 |
| 1 | 12 | -404 | -369 |
| 3 | 12 | -1047 | -1003 |
| 5 | 12 | -316 | -393 |
| 1 | 13 | -642 | -834 |
| 1 | 15 | 308 | 502 |
| L= 6 | | | |
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| 3 | 0 | -994 | -1287 |
| 4 | 0 | 308 | 355 |
| 5 | 0 | -386 | -449 |
| 6 | 0 | 343 | 361 |
| 7 | 0 | -157 | -175 |
| 8 | 0 | 810 | 970 |
| 9 | 0 | 625 | 661 |
| 10 | 0 | 299 | 313 |
| 11 | 0 | 960 | 1055 |
| 12 | 0 | -607 | -629 |
| 13 | 0 | 230 | 263 |
| 2 | 1 | 872 | 819 |
| 3 | 1 | -492 | -494 |
| 4 | 1 | 1312 | 1338 |
| 5 | 1 | 845 | 881 |
| 6 | 1 | 194 | 194 |
| 7 | 1 | 1048 | 1088 |
| 8 | 1 | -430 | -440 |
| 9 | 1 | 361 | 332 |
| 10 | 1 | -712 | -669 |
| 12 | 1 | -554 | -526 |
| 13 | 1 | -686 | -667 |
| 0 | 2 | 449 | 386 |
| 2 | 2 | 458 | 403 |
| 3 | 2 | 475 | 436 |
| 4 | 2 | -237 | -221 |
| 5 | 2 | 634 | 635 |
| 6 | 2 | -457 | -445 |
| 8 | 2 | -360 | -348 |
| 9 | 2 | -615 | -619 |
| 10 | 2 | 203 | 184 |
| 11 | 2 | -210 | -196 |
| 12 | 2 | 264 | 269 |
| 1 | 3 | -131 | -123 |
| 2 | 3 | 194 | 170 |
| 3 | 3 | -210 | -193 |
| 5 | 3 | 308 | 281 |
| 9 | 3 | 229 | 216 |
| 10 | 3 | 203 | 177 |
| 0 | 4 | 1277 | 1079 |
| 1 | 4 | 405 | 354 |
| 2 | 4 | 475 | 412 |
| 3 | 4 | 1074 | 923 |
| 5 | 4 | 784 | 681 |
| 6 | 4 | -906 | -804 |
| 8 | 4 | -871 | -816 |
| 9 | 4 | -871 | -830 |
| 11 | 4 | -545 | -490 |
| 12 | 4 | 537 | 502 |
| 1 | 5 | 1048 | 910 |
| 2 | 5 | -659 | -563 |
| 3 | 5 | 713 | 639 |
| 4 | 5 | -1258 | -1121 |
| 5 | 5 | -1003 | -890 |
| 6 | 5 | -466 | -393 |
| 7 | 5 | -1399 | -1273 |
| 8 | 5 | 696 | 617 |
| 9 | 5 | -228 | -201 |
| 10 | 5 | 537 | 481 |
| 11 | 5 | 255 | 141 |
| 12 | 5 | 599 | 524 |
| 0 | 6 | -1778 | -1541 |
| 1 | 6 | -906 | -803 |
| 2 | 6 | -518 | -410 |
| 3 | 6 | -1390 | -1227 |
| 4 | 6 | 458 | 366 |
| 5 | 6 | -474 | -388 |
| 6 | 6 | 713 | 632 |
| 7 | 6 | -263 | -229 |
| 8 | 6 | 872 | 782 |
| 9 | 6 | 740 | 667 |
| 10 | 6 | 299 | 272 |
| 11 | 6 | 1092 | 945 |
| 12 | 6 | -554 | -476 |
| 1 | 7 | -800 | -735 |
| 2 | 7 | 634 | 550 |
| 4 | 7 | 722 | 620 |
| 5 | 7 | 704 | 625 |
| 6 | 7 | 343 | 271 |
| 7 | 7 | 317 | 294 |
| 8 | 7 | -422 | -393 |
| 9 | 7 | 387 | 352 |
| 10 | 7 | -474 | -436 |
| 0 | 8 | 282 | 211 |
| 1 | 8 | -175 | -174 |
| 5 | 8 | 326 | 324 |
| 6 | 8 | -378 | -350 |
| 1 | 9 | -607 | -551 |
| 2 | 9 | 317 | 318 |
| 4 | 9 | 564 | 586 |
| 5 | 9 | 511 | 462 |
| 7 | 9 | 370 | 329 |
| 8 | 9 | -246 | -182 |
| 10 | 9 | -325 | -299 |
| 0 | 10 | 1013 | 1063 |
| 1 | 10 | 528 | 560 |
| 2 | 10 | 493 | 523 |
| 3 | 10 | 643 | 647 |
| 4 | 10 | -378 | -409 |
| 5 | 10 | 696 | 712 |
| 6 | 10 | -624 | -628 |
| 8 | 10 | -571 | -645 |
| 9 | 10 | -712 | -762 |
| 1 | 11 | 599 | 626 |
| 2 | 11 | -334 | -365 |
| 3 | 11 | 387 | 457 |
| 4 | 11 | -844 | -931 |
| 5 | 11 | -580 | -655 |
| 7 | 11 | -879 | -876 |
| 0 | 12 | -580 | -611 |
| 1 | 12 | -307 | -359 |
| 2 | 12 | -316 | -361 |
| 3 | 12 | -501 | -614 |
| 6 | 12 | 308 | 392 |
| 1 | 15 | -307 | -495 |
| 3 | 16 | 282 | 549 |
| L= 7 | | | |
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| 2 | 1 | -563 | -559 |
| 3 | 1 | -122 | -107 |
| 4 | 1 | -915 | -966 |
| 5 | 1 | 211 | 244 |
| 6 | 1 | -554 | -587 |
| 8 | 1 | 704 | 732 |
| 10 | 1 | 748 | 752 |
| 1 | 2 | 307 | 386 |
| 2 | 2 | -1223 | -1103 |
| 3 | 2 | 132 | 116 |
| 4 | 2 | 423 | 404 |
| 6 | 2 | 1453 | 1452 |
| 7 | 2 | 211 | 214 |
| 8 | 2 | 951 | 933 |
| 9 | 2 | -290 | -310 |
| 11 | 2 | -254 | -245 |
| 12 | 2 | -580 | -561 |
| 1 | 3 | 132 | 88 |
| 2 | 3 | 1770 | 1544 |
| 3 | 3 | 352 | 312 |
| 4 | 3 | 1197 | 1069 |
| 5 | 3 | -360 | -358 |
| 6 | 3 | 440 | 409 |
| 7 | 3 | -325 | -317 |
| 8 | 3 | -290 | -253 |
| 9 | 3 | 238 | 251 |
| 10 | 3 | -1214 | -1178 |
| 12 | 3 | -835 | -774 |
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| 1 | 4 | -157 | -113 |
| 2 | 4 | 766 | 665 |
| 3 | 4 | -193 | -141 |
| 4 | 4 | -281 | -266 |
| 6 | 4 | -1152 | -1033 |
| 8 | 4 | -800 | -733 |
| 11 | 4 | 229 | 109 |
| 12 | 4 | 546 | 480 |
| 1 | 5 | 150 | 152 |
| 4 | 5 | -413 | -373 |
| 5 | 5 | -166 | -177 |
| 6 | 5 | -175 | -130 |
| 8 | 5 | 370 | 353 |
| 0 | 6 | 740 | 657 |
| 2 | 6 | 317 | 268 |
| 4 | 6 | -307 | -267 |
| 6 | 6 | -448 | -389 |
| 8 | 6 | -202 | -172 |
| 12 | 6 | 229 | 167 |
| 2 | 7 | -1047 | -932 |
| 3 | 7 | -219 | -199 |
| 4 | 7 | -1355 | -1202 |
| 6 | 7 | -483 | -456 |
| 7 | 7 | 211 | 144 |
| 8 | 7 | 643 | 592 |
| 10 | 7 | 1206 | 1017 |
| 0 | 8 | -818 | -745 |
| 1 | 8 | 379 | 356 |
| 2 | 8 | -932 | -889 |
| 6 | 8 | 1259 | 1166 |
| 7 | 8 | 203 | 130 |
| 8 | 8 | 933 | 844 |
| 2 | 9 | 1057 | 1051 |
| 3 | 9 | 247 | 246 |
| 4 | 9 | 836 | 772 |
| 6 | 9 | 299 | 282 |
| 8 | 9 | -281 | -231 |
| 10 | 9 | -888 | -724 |
| 0 | 10 | 484 | 517 |
| 2 | 10 | 247 | 218 |
| 4 | 10 | -281 | -273 |
| 2 | 11 | 229 | 246 |
| 0 | 12 | 634 | 751 |
| 2 | 12 | 335 | 431 |
| 0 | 14 | -281 | -602 |
| L= 8 | | | |
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| 2 | 0 | 255 | 309 |
| 3 | 0 | -1311 | -1249 |
| 4 | 0 | -395 | -477 |
| 5 | 0 | -695 | -738 |
| 6 | 0 | -360 | -369 |
| 7 | 0 | 352 | 385 |
| 8 | 0 | -430 | -464 |
| 9 | 0 | 519 | 510 |
| 10 | 0 | -272 | -224 |
| 11 | 0 | 616 | 604 |
| 12 | 0 | 405 | 385 |
| 13 | 0 | 387 | 339 |
| 1 | 1 | -774 | -993 |
| 2 | 1 | -404 | -480 |
| 4 | 1 | -747 | -838 |
| 5 | 1 | 396 | 426 |
| 6 | 1 | -166 | -159 |
| 7 | 1 | 801 | 864 |
| 8 | 1 | 264 | 272 |
| 9 | 1 | 555 | 567 |
| 10 | 1 | 564 | 577 |
| 11 | 1 | -316 | -336 |
| 12 | 1 | 291 | 277 |
| 13 | 1 | -695 | -557 |
| 0 | 2 | 150 | 119 |
| 2 | 2 | -272 | -340 |
| 3 | 2 | 185 | 185 |
| 5 | 2 | 431 | 476 |
| 6 | 2 | 264 | 273 |
| 8 | 2 | 247 | 277 |
| 9 | 2 | -263 | -264 |
| 11 | 2 | -219 | -153 |
| 1 | 3 | -131 | -113 |
| 2 | 3 | -131 | -99 |
| 4 | 3 | -166 | -141 |
| 9 | 3 | 211 | 173 |
| 10 | 3 | 194 | 127 |
| 0 | 4 | -563 | -658 |
| 2 | 4 | -334 | -349 |
| 3 | 4 | 643 | 701 |
| 5 | 4 | 696 | 722 |
| 6 | 4 | 608 | 640 |
| 7 | 4 | -193 | -185 |
| 8 | 4 | 475 | 478 |
| 9 | 4 | -413 | -408 |
| 11 | 4 | -360 | -360 |
| 12 | 4 | -325 | -273 |
| 1 | 5 | 889 | 961 |
| 2 | 5 | 387 | 415 |
| 4 | 5 | 608 | 664 |
| 5 | 5 | -466 | -487 |
| 6 | 5 | 370 | 369 |
| 7 | 5 | -765 | -824 |
| 8 | 5 | -510 | -531 |
| 9 | 5 | -510 | -552 |
| 10 | 5 | -334 | -319 |
| 11 | 5 | 449 | 429 |
| 12 | 5 | -360 | -331 |
| 0 | 6 | 660 | 841 |
| 1 | 6 | -307 | -349 |
| 2 | 6 | 299 | 247 |
| 3 | 6 | -862 | -1068 |
| 4 | 6 | -246 | -225 |
| 5 | 6 | -615 | -728 |
| 6 | 6 | -325 | -336 |
| 7 | 6 | 211 | 233 |
| 8 | 6 | -386 | -459 |
| 9 | 6 | 493 | 585 |
| 1 | 7 | -466 | -589 |
| 2 | 7 | -334 | -372 |
| 4 | 7 | -246 | -211 |
| 5 | 7 | 335 | 322 |
| 7 | 7 | 335 | 314 |
| 9 | 7 | 291 | 298 |
| 0 | 8 | 194 | 87 |
| 4 | 8 | -228 | -230 |
| 1 | 9 | -510 | -507 |
| 2 | 9 | -386 | -344 |
| 3 | 9 | 273 | 165 |
| 4 | 9 | -404 | -328 |
| 0 | 10 | -474 | -428 |
| 2 | 10 | -281 | -256 |
| 3 | 10 | 431 | 601 |
| 1 | 11 | 669 | 721 |
| 0 | 12 | 238 | 273 |
| 1 | 12 | -281 | -271 |
| 3 | 12 | -483 | -561 |

Table 3. *Final parameters for the atoms of* $\alpha$-Ba[AlO(OH)$_2$]$_2$

Figures in brackets represent the estimated standard deviation corresponding to the least significant digit.

| | Coordinates (fractions of cell edge) | | | Isotropic temperature factor $B$ |
|---|---|---|---|---|
| | $x$ | $y$ | $z$ | (Å$^2$) |
| Ba | 0·0733 (1) | 0·3433 (1) | 0·3947 (1) | 1·54 (2) |
| Al(1) | 0·149 (1) | 0·015 (1) | 0·387 (1) | 1·1 (1) |
| Al(2) | 0·326 (1) | 0·150 (1) | 0·226 (1) | 1·1 (1) |
| O(1) | 0·429 (1) | 0·222 (1) | 0·328 (2) | 1·4 (2) |
| O(2) | 0·094 (1) | −0·008 (1) | 0·162 (2) | 1·0 (1) |
| O(3) | 0·223 (1) | 0·242 (1) | 0·180 (2) | 1·7 (2) |
| O(4) | 0·0795 (10) | 0·120 (1) | 0·493 (2) | 1·3 (2) |
| O(5) | 0·278 (1) | 0·053 (1) | 0·373 (2) | 1·4 (2) |
| O(6) | 0·370 (1) | 0·103 (1) | 0·017 (2) | 1·1 (2) |

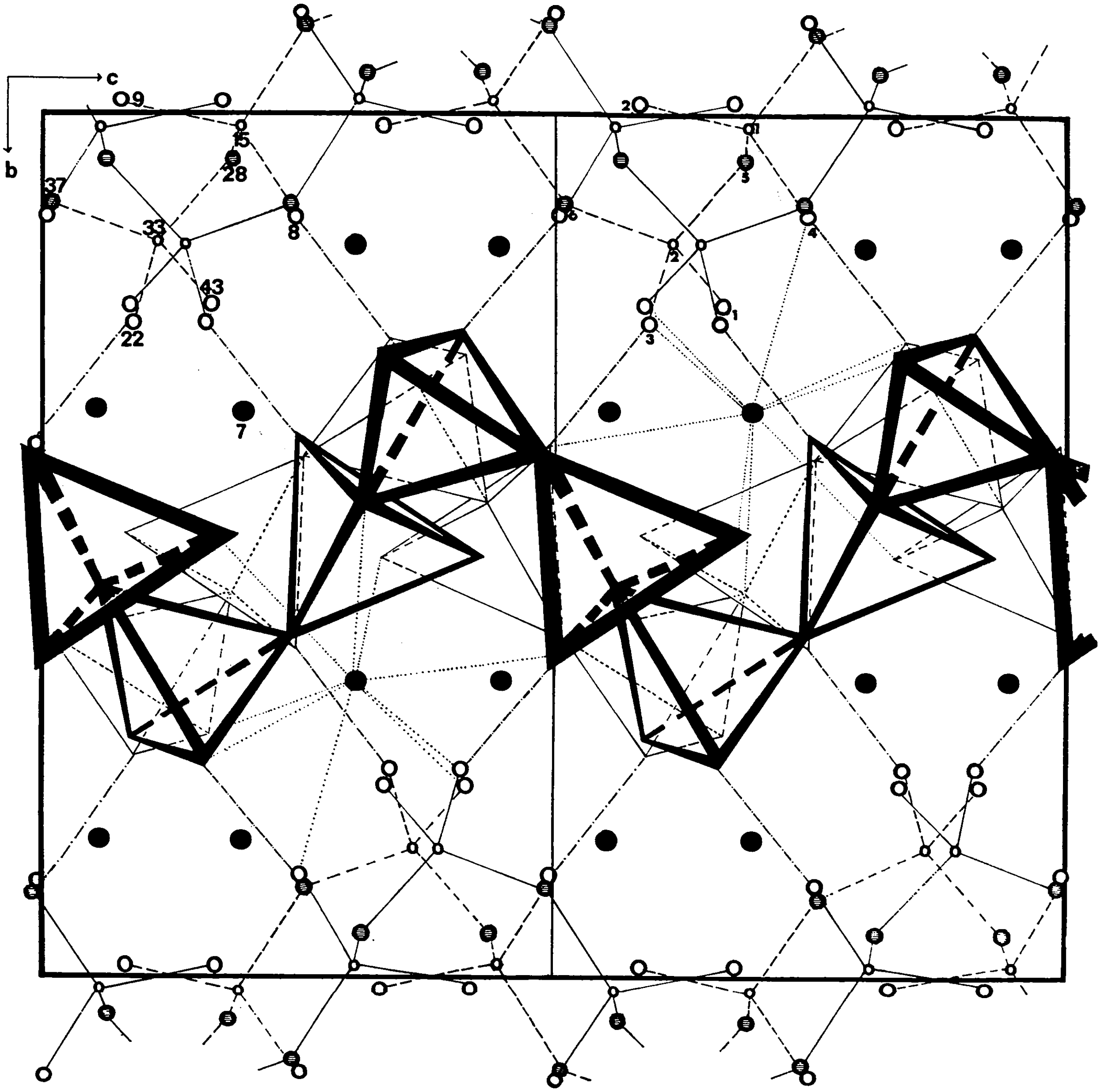
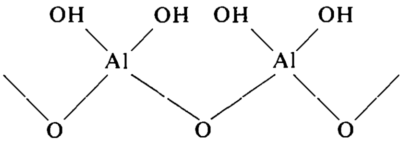


Fig. 1. Projection of two cells of $\alpha$-Ba[AlO(OH)$_2$]$_2$ on (100), showing the configuration of the [AlO(OH)$_2$]$_\infty$ chains (centre). Large solid circles represent barium atoms and small open circles aluminum atoms. Medium circles represent oxygen atoms (shaded) and hydroxyl groups (open). The larger figures (left hand cell) give heights in $a$/100; the small figures in the right hand cell give atom designations for the appropriate asymmetric unit (*cf.* Table 3). Dot-dash lines represent hydrogen bonds.

the region immediately adjacent to the barium atom where the values were higher, probably reflecting the fact that isotropic temperature factors only had been used in the refinement. No indication of the hydrogen positions could be obtained.

## Description of the structure

Fig. 1 shows the structure projected on (100). Each aluminum atom has as nearest neighbours four oxygen atoms disposed at the corners of a tetrahedron. The tetrahedra are joined together to form single helical chains running along the $2_1$ axes parallel to **c**. The 'water' is thus presumably all present as hydroxyl groups and the anion can be written $[AlO(OH)_2^-]_\infty$. Each tetrahedron has two types of oxygen atoms, *viz.* (1) bridging oxygen atoms, and (2) non-bridging oxygen atoms. Each of the latter is presumably associated with a hydrogen atom giving the anion



The single chains (in which Al(1)-oxygen tetrahedra alternate with Al(2)-oxygen tetrahedra) are linked by the barium atoms, each of which is coordinated by eight non-bridging oxygen (hydroxyl) atoms at distances in the range 2·78–3.02 Å and one bridging oxygen [O(5)] only slightly more remote than this (3·22 Å). The configuration of the $BaO_9$ polyhedron may be described as follows. Six of the coordinating oxygen atoms (including the most remote one) form a distorted triangular prism with the barium at its centre; the remaining three lie just outside the centres of the prism faces.

Study of the O–O bond distances suggests that the chains having the same *x* coordinates are linked by H bonds [O(3) – O(4′)], some of which are shown in Fig. 1.

The [Al(2) – O] tetrahedra are more distorted than the [Al(1) – O] tetrahedra (Table 4). It is by no means clear why this is. The mean of the distances from Al to non-bridging oxygen atoms (1·77 Å) is somewhat longer than that to the bridging ones (1·74 Å) but the scatter is such that it is difficult to say whether this is significant. Each OH group is linked to two barium atoms and one aluminum atom; each of the bridging oxygen atoms is linked to two aluminum atoms and one of them also makes contact with one barium atom. In order to apply Pauling's electrostatic valency rule, as a first approximation the bonds between the barium and bridging oxygen atoms may be neglected. This leads to the distribution of electrostatic bond strengths shown below:

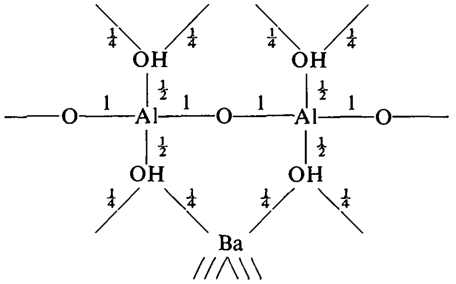


Table 4. *Interatomic distances and bond angles*

Figures in brackets represent the estimated standard deviation corresponding to the least significant digit. O(6′) is related to O(6) by the $2_1$ axis parallel to **c**. Other atoms in different asymmetric units are indicated by letters, which represent the operation of glide planes on the original atoms. Thus, O(2*a* + *c*) is derived from O(2) by an *a*-glide translation followed by a *c*-glide translation.

*A*. Al(1)–O tetrahedron

| Interatomic distances (Å) | | Bond angles (e.s.d. 1°) | |
|---|---|---|---|
| Al(1)–O(2) | 1·78 (2) | O(2)–Al(1)–O(4) | 108·3° |
| Al(1)–O(4) | 1·75 (2) | O(2)–Al(1)–O(5) | 111·8 |
| Al(1)–O(5) | 1·75 (2) | O(2)–Al(1)–O(6′) | 107·2 |
| Al(1)–O(6′) | 1·75 (2) | O(4)–Al(1)–O(5) | 108·8 |
| O(2) –O(4) | 2·87 (2) | O(4)–Al(1)–O(6′) | 108·2 |
| O(2) –O(5) | 2·92 (2) | O(5)–Al(1)–O(6′) | 112·5 |
| O(2) –O(6′) | 2·85 (2) | | |
| O(4) –O(5) | 2·84 (2) | | |
| O(4) –O(6′) | 2·84 (2) | | |
| O(5) –O(6′) | 2·91 (2) | | |

*B*. Al(2)–O tetrahedron

| Interatomic distances (Å) | | Bond angles (e.s.d. 1°) | |
|---|---|---|---|
| Al(2)–O(1) | 1·77 (2) | O(1)–Al(2)–O(3) | 109·1° |
| Al(2)–O(3) | 1·78 (2) | O(1)–Al(2)–O(5) | 111·9 |
| Al(2)–O(5) | 1·72 (2) | O(1)–Al(2)–O(6) | 106·3 |
| Al(2)–O(6) | 1·71 (2) | O(3)–Al(2)–O(5) | 106·7 |
| O(1) –O(3) | 2·89 (2) | O(3)–Al(2)–O(6) | 107·5 |
| O(1) –O(5) | 2·89 (2) | O(5)–Al(2)–O(6) | 115·2 |
| O(1) –O(6) | 2·79 (2) | | |
| O(3) –O(5) | 2·80 (2) | | |
| O(3) –O(6) | 2·81 (2) | | |
| O(5) –O(6) | 2·90 (2) | | |

Table 4 (*cont.*)

| | |
|---|---|
| *C*. Ba–O polyhedron | |
| Interatomic distances (Å) | |
| Ba–O(1*a*+*c*) | 2·85 (1) |
| Ba–O(3*c*) | 3·02 (1) |
| Ba–O(1*a*) | 2·88 (1) |
| Ba–O(3) | 2·78 (1) |
| Ba–O(4) | 2·85 (1) |
| Ba–O(2*c*) | 2·81 (1) |
| Ba–O(2*a*+*c*) | 2·88 (1) |
| Ba–O(4*c*) | 2·92 (1) |
| Ba–O(5*b*) | 3·22 (1) |
| *D*. Hydrogen bond | |
| O(4)–O(3*c*) | 2·86 (1) Å |

A more satisfactory distribution might be obtained by assigning some arbitrary small value to the bonds between barium and bridging oxygen atoms; this would tend to equalize the electrostatic bond strengths of the Al–O bonds and to reduce slightly those of the remaining Ba–OH bonds.

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# The Crystal and Molecular Structure of a Hydrazone Derivative of 2,2′-Di-(1,4-naphthoquinone)

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The title compound, $C_{21}H_{12}N_2O_3$, crystallizes in the orthorhombic space group *Pbca* with unit-cell dimensions of $a=14·45$, $b=8·22$ and $c=25·58$ Å, and with eight molecules per unit cell. Three-dimensional X-ray diffraction data were recorded on equi-inclination Weissenberg films using Cu $K\alpha$ radiation, and the 2196 intensities were visually estimated. The structure was solved with the symbolic addition method and has been refined with full-matrix least-squares to an $R$ index of 0·10; anisotropic temperature factors were used for the carbon, nitrogen and oxygen atoms and the hydrogen atoms were included with isotropic temperature factors. The molecule is reasonably planar and structural requirements have produced two close intramolecular contacts, O···H and $CH_3$···O. Bond distances in the heterocyclic portion of the molecule show the effects of $\pi$-electron delocalization.

## Introduction

The reaction of 1,4-naphthoquinone (I) with hydrazine hydrate does not occur in the usual way to yield the familar ketonic hydrazone derivative. Hand & Cohen (1967) have reported that the naphthoquinone 'hydrazone' and the compound isolated from the reaction of hydrazine hydrate with 2,2′-di-(1,4-naphthoquinone) (II) are identical, and they have shown that the material is the cinnoline derivative (III*a*). Our investigation of the structure of (III) was stimulated by some recent

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