Table 2. Interatomic distances and angles for euchroite

| Within the $\mathrm{Cu}(1)$ polyhedron |  |  | Within the $\mathrm{Cu}(2)$ polyhedron |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  | (F) | (G) |  | (F) | (G) |
| $\mathrm{Cu}(1)-\mathrm{O}(4)$ | 1.983 | $1 \cdot 92$ | $\mathrm{Cu}(2)-\mathrm{O}(1)$ | 1.946 | 1.99 |
| -O(5) | 1.963 | 2.08 | -O(2) | 2.795 | 2.74 |
| -O(6) | 2.365 | $2 \cdot 42$ | -O(3) | 1.958 | 1.97 |
| -O(6') | 2.479 | 2.51 | -O(4) | 2.397 | $2 \cdot 47$ |
| -O(7) | 1.943 | 1.96 | -O(5) | 1.989 | 1.92 |
| $-\mathrm{O}\left(7^{\prime}\right)$ | 2.044 | 2.01 | -O(7) | 2.008 | 2.01 |
| $\mathrm{O}(4)-\mathrm{Cu}(1)-\mathrm{O}(6)$ | $89.5{ }^{\circ}$ | $90^{\circ}$ | $\mathrm{O}(1)-\mathrm{Cu}(2)-\mathrm{O}(2)$ | $84.5{ }^{\circ}$ | $86^{\circ}$ |
| $\mathrm{O}(4)-\mathrm{Cu}(1)-\mathrm{O}\left(6^{\prime}\right)$ | $93 \cdot 1$ | 93 | $\mathrm{O}(1)-\mathrm{Cu}(2)-\mathrm{O}(3)$ | $93 \cdot 9$ | 93 |
| $\mathrm{O}(4)-\mathrm{Cu}(1)-\mathrm{O}(7)$ | $86 \cdot 2$ | 88 | $\mathrm{O}(1)-\mathrm{Cu}(2)-\mathrm{O}(4)$ | $98 \cdot 6$ | 98 |
| $\mathrm{O}(4)-\mathrm{Cu}(1)-\mathrm{O}\left(7^{\prime}\right)$ | $97 \cdot 1$ | 96 | $\mathrm{O}(1)-\mathrm{Cu}(2)-\mathrm{O}(5)$ | $92 \cdot 1$ | 90 |
| $\mathrm{O}(5)-\mathrm{Cu}(1)-\mathrm{O}(6)$ | $86 \cdot 9$ | 88 | $\mathrm{O}(2)-\mathrm{Cu}(2)-\mathrm{O}(3)$ | $87 \cdot 1$ | 87 |
| $\mathrm{O}(5)-\mathrm{Cu}(1)-\mathrm{O}\left(6^{\prime}\right)$ | $90 \cdot 3$ | 89 | $\mathrm{O}(2)-\mathrm{Cu}(2)-\mathrm{O}(5)$ | 82.4 | 79 |
| $\mathrm{O}(5)-\mathrm{Cu}(1)-\mathrm{O}(7)$ | $95 \cdot 5$ | 96 | $\mathrm{O}(2)-\mathrm{Cu}(2)-\mathrm{O}(7)$ | $102 \cdot 6$ | 102 |
| $\mathrm{O}(5)-\mathrm{Cu}(1)-\mathrm{O}\left(7^{\prime}\right)$ | 81.6 | 80 | $\mathrm{O}(3)-\mathrm{Cu}(2)-\mathrm{O}(4)$ | 92.2 | 94 |
| $\mathrm{O}(6)-\mathrm{Cu}(1)-\mathrm{O}(7)$ | $95 \cdot 0$ | 95 | $\mathrm{O}(3)-\mathrm{Cu}(2)-\mathrm{O}(7)$ | 93.6 | 95 |
| $\mathrm{O}(6)-\mathrm{Cu}(1)-\mathrm{O}\left(7^{\prime}\right)$ | 89.8 | 90 | $\mathrm{O}(4)-\mathrm{Cu}(2)-\mathrm{O}(5)$ | $74 \cdot 4$ | 73 |
| $\mathrm{O}\left(6^{\prime}\right)-\mathrm{Cu}(1)-\mathrm{O}(7)$ | 89.0 | 89 | $\mathrm{O}(5)-\mathrm{Cu}(2)-\mathrm{O}(7)$ | 81.8 | 84 |
| $\mathrm{O}(4)-\mathrm{Cu}(1)-\mathrm{O}(5)$ | $176 \cdot 2$ | - | $\mathrm{O}(1)-\mathrm{Cu}(2)-\mathrm{O}(7)$ | $170 \cdot 0$ | - |
| $\mathrm{O}(6)-\mathrm{Cu}(1)-\mathrm{O}\left(6^{\prime}\right)$ | $175 \cdot 3$ | - | $\mathrm{O}(2)-\mathrm{Cu}(2)-\mathrm{O}(4)$ | 176.9 | - |
| $\mathrm{O}(7)-\mathrm{Cu}(1)-\mathrm{O}\left(7^{\prime}\right)$ | $174 \cdot 2$ | - | $\mathrm{O}(3)-\mathrm{Cu}(2)-\mathrm{O}(5)$ | $167 \cdot 3$ | - |
| Within the arsenate tetrahedron |  |  |  |  |  |
| As-O(3) | 1.680 | 1.68 | $\mathrm{O}(3)-\mathrm{As}-\mathrm{O}(4)$ | $104.8^{\circ}$ | $103^{\circ}$ |
| -O(4) | 1.664 | 1.65 | $\mathrm{O}(3)-\mathrm{As}-\mathrm{O}(5)$ | $109 \cdot 3$ | 111 |
| -O(5) | 1.773 | 1.73 | $\mathrm{O}(3)-\mathrm{As}-\mathrm{O}(8)$ | $110 \cdot 1$ | 112 |
| -O(8) | 1.615 | 1.61 | $\mathrm{O}(4)-\mathrm{As}-\mathrm{O}(5)$ | 109.9 | 109 |
|  |  |  | $\mathrm{O}(4)-\mathrm{As}-\mathrm{O}(8)$ | $114 \cdot 4$ | 116 |
| Hydrogen bond distances |  |  | $\mathrm{O}(5)-\mathrm{As}-\mathrm{O}(8)$ | 108.3 | 105 |
| $\mathrm{O}(1)-\mathrm{O}(6)$ | 3.052 | 2.95 |  |  |  |
| $\mathrm{O}(1)-\mathrm{O}(8)$ | 2.583 | 2.61 |  |  |  |
| $\mathrm{O}\left(1^{\prime}\right)-\mathrm{O}(8)$ | 2.834 | 2.77 |  |  |  |
| $\mathrm{O}(2)-\mathrm{O}(6)$ | 2.753 | $2 \cdot 94$ |  |  |  |
| $\mathrm{O}\left(2^{\prime}\right)-\mathrm{O}(6)$ | 2.882 | 2.70 |  |  |  |
| $\mathrm{O}(7)-\mathrm{O}(8)$ | 2.585 | $2 \cdot 63$ |  |  |  |
| $\mathrm{O}(2)-\mathrm{O}(5)$ | 2.918 | $2 \cdot 96$ |  |  |  |

The average standard deviations of the bond lengths for this refinement are: $\mathrm{Cu}-\mathrm{O} 0.012$; As-O 0.012 . That of the angles is $0.50^{\circ}$.
molecules. The difference in birefringence may be partly caused by the rotation of the structural unit into a plane parallel to (010) in euchroite.

It has been stated (Palache, Berman \& Frondel, 1951) that euchroite possesses a $\{110\}$ and $\{101\}$ cleavage in traces. Fig. 1 shows the hypothesized $\{110\}$ cleavage developed by breaking hydrogen bonds between $O(1)$ and $O(8)$ and between $O(7)$ and $O(8)$. Also one stronger bond need be broken between $\mathrm{Cu}(2)$ and $\mathrm{O}(3)$. The $\{101\}$ cleavage cannot be accounted for in any simple manner.

The author wishes to thank Dr L. G. Berry for the crystals of euchroite. R.P.Sage collected the molybdenum data. The Colorado School of Mines Foundation, Inc. grant 6402 provided the author with funds for the completion of this
work. Especial thanks are due the University of Colorado Computing Center for computing time and technical assistance.

## References

Berry, L. G. (1951). Amer. Min. 36, 496.
Cromer, D. T., Larson, A. C. \& Waber, J. T. (1963). Los Alamos Scientific Laboratory Report LA-2987.
Giuseppetti, G. (1963). Period. Mineral. 32, 131.
Heritsch, H. (1938). Z. Kristallogr. 99, 466.
Heritsch, H. (1940). Z. Kristallogr. 102, 1.
Palache, C., Berman, H. \& Frondel, C. (1951). Dana's System of Mineralogy, Vol. II, p.934. New York: John Wiley.
Walitzi, E. M. (1963). Miner. Petrog. Mitt. 8, 614.

Acta Cryst. (1966). 21, 440
The similarity of atomic radius of palladium (II) and palladium (IV). By J.D.Bell, D. Hall and T. N. Waters,
Chemistry Department, University of Auckland, New Zealand
(Received 28 February 1966)

A number of apparently trivalent complexes of palladium and platinum have been investigated crystallographically (Brosset, 1948; Cohen \& Hughes, 1954; Hall \& Williams,

1958; Craven \& Hall, 1961, 1966; Ryan \& Rundle, 1961 ; Wallen, Brosset \& Vannerberg, 1962), and invariably the structures have proved to be based on chains in which
divalent and tetravalent atoms alternate, e.g. $\mathrm{Pd}\left(\mathrm{NH}_{3}\right)_{2} \mathrm{Cl}_{3}$ is in fact $\mathrm{Pd}^{\mathrm{II}}\left(\mathrm{NH}_{3}\right)_{2} \mathrm{Cl}_{2} . \mathrm{Pd}^{\mathrm{IV}}\left(\mathrm{NH}_{3}\right)_{2} \mathrm{Cl}_{4}$, with structure


In practice, however, the diffraction patterns show evidence of disorder, the stacking of chains being susceptible to mistakes which involve their translation by one half the repeat distance in the chain direction. It has been supposed that this is possible because the dimensions of the complex ions are essentially the same, irrespective of the valence state of the central metal, and this view is supported by the atomic radii listed by Pauling (1960). Nonetheless there does not appear to be any instance in which bond lengths have been measured in comparable compounds with sufficient precision to make such an assertion, and for this reason the well established structures of ammonium chloropalladate(II) and ammonium chloropalladate(IV) have been re-examined.

## Ammonium chloropalladate (II)

Crystals of $\left(\mathrm{NH}_{4}\right)_{2} \mathrm{PdCl}_{4}$ were grown as fine bronze tetragonal needles which were strongly pleochroic. Cell dimensions were determined from Weissenberg photographs by the method of Main \& Woolfson (1963) as $a=7 \cdot 205 \pm$ $0.006, c=4.26 \pm 0.02 \AA$, in agreement with the values previ-
ously reported by Dickinson (1922). The density was measured by flotation as $2 \cdot 1 \mathrm{~g} . \mathrm{cm}^{-3}$, and the calculated value for one molecule per unit cell is $2 \cdot 14$. Intensities were measured visually from Weissenberg photographs, taken with $\mathrm{Cu} K \alpha$ radiation, for the layers $h k 0-h k 3$. The crystals used were of square section, and cylindrical absorption corrections were applied. The $h 0 l$ layer was also photographed, using a crystal which had been cut to an approximate cube. It did not, however, prove possible to obtain such a crystal that was not multiple, and the $h 0 l$ data were used to correlate the layers about a, but were otherwise excluded from the refinement.

The structure was confirmed as that previously described (Dickinson, 1922) i.e. as in $P 4 / m m m$, with palladium in (a), $0,0,0$; nitrogen in (e), $0, \frac{1}{2}, \frac{1}{2}$; and chlorine in $(j), x, x, 0$, with $x \sim 0 \cdot 23$. The chlorine parameter $x$, anisotropic thermal parameters for palladium and chlorine, and an isotropic thermal parameter for nitrogen, were then refined by a block-diagonal least-squares procedure. Scattering curves assumed were those of Thomas \& Umeda (1957) for palladium and those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry \& Veenendaal (1955) for chlorine and nitrogen. Real corrections for dispersion were made to the palladium and chlorine values. The weighting system was similar to the Hughes scheme except that terms for very small $F_{\text {obs }}$ were further downweighted. Thirteen terms which appeared to be affected by extinction were removed from the data. The final $R$ index for the 118 independent terms included was 0.073 . The final values for the parameters are listed in Table 1 and observed and calculated

Table 1. Atomic parameters

| Ammonium chloropalladate(II) |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | $x$ | $y$ | $z$ | $b_{11}$ | $b_{12}$ | $b_{33}$ | B |
| Pd | 0 | 0 | 0 | $0 \cdot 00523$ | - | 0.02868 |  |
| Cl | 0.2257 | $0 \cdot 2257$ | 0 | $0 \cdot 01212$ | -0.00543 | 0.06072 |  |
| N | 0 | $\frac{1}{2}$ | $\frac{1}{2}$ |  |  |  | $4 \cdot 28$ |
| Ammonium chloropalladate(IV) |  |  |  |  |  |  |  |
| Pd | 0 | 0 | 0 |  |  |  | 0.413 |
| Cl | 0.2337 | 0 | 0 | $0 \cdot 00254$ | - | $0 \cdot 00516$ |  |
| N | , | $\frac{1}{4}$ |  |  |  |  | $2 \cdot 47$ |

Table 2. Observed and calculated structure factors $(\times 2.5)$
Values marked with an asterisk were assumed to be affected by extinction. The table gives, reading from left to right, $h, k, l, F_{o}, F_{c}$.

Ammonium chloropalladate (II)

| 1 | 1 | - | 963 | 1238 | 6 | 40 | 421 | 481 | 3 | 3 | 1370 | 4413 | 8 | 1 | 1 | 700 | 636 | 5 | 2 | 2 | 261 | 299 | 2 | 2 | 3 | 1415 | 1337 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 2 | 0 | 0 | 148 | -115 | 6 | 50 | 357 | 355 | 4 | 01 | 1660* | 1923 | 8 | 2 | 1 | 397 | 401 | 5 | 3 | 2 | 495 | 502 | 3 | 0 | 3 | 551 | 479 |
| 2 | 1 | 0 | 1096 | 1204 | 6 | 60 | 839 | 725 | 4 | 11 | 1254 | 1253 | 8 | 3 | 1 | 485 | 433 | 5 | 4 |  | 980 | 1029 | 3 | 1 | 3 | 941 | 828 |
| 2 | 2 | 0 | 1811* | 3348 | 7 | 10 | 611 | 599 | 4 | 31 | 665 | 577 | 8 | 4 | 1 | 548 | 556 | 5 | 5 | 2 | 910 | 71 | 3 | 2 | 3 | 1069 | 1023 |
| 3 | 0 | 0 | 620 | 599 | 7 | 0 | 1101 | 1246 | 4 | 41 | 1211 | 1328 | 9 | 0 | 1 | 714 | 870 | 6 | 0 | 2 | 406 | 394 | 3 | 3 | 3 | 914 | 860 |
| 3 | 1 | 0 | 982 | 966 | 7 | 30 | 919 | 321 | 5 | 01 | 1600 | 1636 | 9 | 1 | 1 | 454 | 571 | 6 | 1 | 2 | 666 | 633 | 4 | 0 | 3 | 1096 | 1146 |
| 3 | 2 | 0 | 1477 | 1890 | 8 | 0 | 949 | 843 | 5 | 1 | 1174 | 1145 | 1 | 0 | 2 | 979* | 1409 | 6 | 2 | 2 | 1131 | :069 | 4 | 1 | 3 | 821 | 790 |
| 3 | 3 | 0 | 1135 | 1158 | 8 | 0 | 812 | 666 | 5 | 21 | 316 | 306 | 1 |  | 2 | 898 | 969 | 6 | 3 | 2 | 852 | 827 | 4 | 3 | 3 | 434 | 435 |
| 4 | $\bigcirc$ | 0 | 1945* | 2491 | 8 | 20 | 542 | 483 | 5 | 31 | 764 | 727 | 2 | - | 2 | 254 | 161 | 6 | 4 | 2 | 433 | 420 | 4 | 4 | 3 | 904 | 847 |
| 4 | 1 | 0 | 1262 | 1342 | 8 | 3 c | 488 | 4.8 | 5 | 41 | 114. | 1224 | 2 | 1 | 2 | $875^{\circ}$ | 935 | 6 | 5 | 2 | 222 | 324 | 5 | 0 | 3 | 1 C 15 | 1004 |
| 4 | 2 | 0 | 354 | 339 | 8 | 40 | 665 | 633 | 5 | 51 | 1 C 74 | 1005 | 2 | 2 | 2 | 1752* | 2226 | 6 | 6 | - | 54.5 | 596 | 5 | 1 | 3 | 736 | 733 |
| 4 | 3 | 0 | 595 | 598 |  | 0 | 894 | 920 | 6 | 01 | 339 | 266 | 3 | , | 2 | 578 | 565 | 7 | , | 2 | 558 | 509 | 5 | 2 | 3 | 275 | 285 |
| 4 | 4 | 0 | $1416 *$ | 1612 | 9 | 10 | 545 | 550 | 6 | 11 | 748 | 725 | 3 | 1 | 2 | 765 | 810 | 7 | 2 | 2 | 1052 | 991 | 5 | 3 | 3 | 569 | 508 |
| 5 | c | $\bigcirc$ | $1624^{\circ}$ | 1752 | 1 | 1 | 1078** | 1873 | 6 | 21 | 1171 | 1136 | 3 | 2 | 2 | ${ }^{1240 *}$ | 1392 | 7 | 3 | 2 | 722 | 675 | 5 | 4 | 3 | 776 | 78.4 |
| 5 | 1 | c | 1 C 20 | 964 | 1 | 11 | $1354^{\circ}$ | 1924 | 6 | 31 | 338 | 971 | 3 | 3 | 2 | 958 | 928 | 7 | 5 | 2 | 155 | 266 | 5 | 5 | 3 | 660 | 662 |
| 5 | 2 | 0 | 256 | 304 | 2 | 01 | 787 | -690 | 6 | 1 | 329 | 347 | 4 |  | 2. | 1584* | 1797 | 8 | 0 | 2 | 740 | 694 | 6 | 1 | 3 | 559 | 509 |
| 5 | 3 | 0 | 552 | 576 | 2 | 11 | $1{ }^{1} 4$ | 1120 | 6 | 61 | 777 | 633 | 4 |  | 2 | 939 | 1039 | 8 | 1 | 2 | 459 | 557 | 6 | 2 | 3 | 770 | 737 |
| 5 | 4 | 0 | 1119 | 1299 | 2 | 21 | 1525* | 24.5 | 7 | 1 | 734 | 678 | 4 | 2 | 2 | 354 | 334 | 8 | 2 | 2 | 388 | 417 | 6 | 3 | 3 | 613 | 64.4 |
| 5 | 5 | 0 | 975 | 951 | 3 | 01 | 685 | 598 | 7 | 21 | 1203 | 1176 | 4 | 3 | 2 | 549 | 513 | 8 | 3 | 2 | 323 | 389 | 6 | 4 | 3 | 225 | 303 |
| 6 | 0 | 0 | 4.4 | 439 | 3 | 11 | 1275 | 1400 | 7 | 3.1 | 980 | 864 | 4 | 8 | 2 | . 1214 | 1256 | 1 | c | 3 | 866 | 1003 | 7 | 1 | 3 | 453 | 478 |
| 6 | 1 | 0 | 783 | 760 | 3 | 21 | 1539 | 1740 | 7 | 5 '1 | 315 | 335 | 5 | 0 | 2 | 1274 | 13,49 | 1 | 1 | 3 | 1049 | 1001 | 7 | 2 | 3 | 696 |  |
| 6 | 2 | 0 | 126. | 1355 |  |  |  |  | 8 | 01 | 821 | 731 | 5 | 1 | 2 | 812 | 791 | 2 | 1 | 3 | 834 | 719 | 7 | 3 | 3 | 492 | 578 |
| 6 | 3 | 0 | 945 | 1 C 26 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

Ammonium chloropalladate (IV)

| c 2 | 0 | 3461 | 4954 | 6 |  | c | 3015 | 2967 | 6 | 8 | 0 | 3684 | 3141 | 12 | 2 | 0 | 1605 | 1528 | 5 | 3 | 1 | 2756 | 2806 | 9 | 1 | 1 | 2194 | 2787 |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 2 | 0 | 1905 | 134, 2 | 6 | 6 | 0 | 1525 | 1420 | 10 | 0 | 0 | 2313 | 2462 | 12 | 4. | 0 | 3247 | 2541 | 5 | 5 | 1 | 3465 | 3392 | 9 | 3 | 1 | 2312 | 2342 |
| 40 | - | 5691 | 8303 | 8 | 0 | 0 | 3127 | 4929 | 10 | 2 | 0 | 1299 | 1334 | 1 | 1 | 1 | 2666 | 4178 | 7 | 1 | 1 | 1203 | 1564 |  | 5 | 1 | 2945 | 2718 |
| 42 | 0 | 3127 | 3735 | 8 | 2 | - | 2274 | 2470 | 10 | 4 | 0 | 234.5 | 2234 | 3 | 1 | 1 | $2 ? 69$ | 2797 | 7 | 3 | 1 | 1118 | 1094 | 9 | 7 | 1 | 1780 | 1666 |
| 44 | c | 54.63 | 6950 | 8 | 4 | 0 | 4350 | 4370 | 10 | 6 | 0 | 1221 | 1241 | 3 | 3 | 1 | 1549 | 1872 | 7 | 5 | 1 | 2037 | 1774 | 11 | 1 | 1 | 887 | 388 |
| 60 | 0 | 2538 | 2398 | $\bigcirc$ | 6 | c | 2592 | 2164 | 12 | 0 | 0 | 2820 | 2806 | 5 | 1 | 1 | 2:15 | 3620 | 7 | 7 | 1 | 819 | 672 | 11 | 5 | 1 | 922 | 1075 |
| 62 | c | 1512 | : 603 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

structure factors are listed in Table 2. The palladiumchlorine bond length is $2.299 \AA$, with $\sigma=0.004 \AA$. This estimate of error allows for uncertainty in both atomic parameter and cell dimension.

## Ammonium chloropalladate(IV)

Crystals of $\left(\mathrm{NH}_{4}\right)_{2} \mathrm{PdCl}_{6}$ were obtained, following Sharpe (1953), as small wine-red octahedra. The axial length of the cubic cell was determined from a calibrated rotation photograph as $9.84 \pm 0.01 \AA$, in agreement with Sharpe (1953). The density has been measured as $2.48 \mathrm{~g} . \mathrm{cm}^{-3}$ (Ketelaar \& von Walsem, 1938), and the calculated volume for 4 molecules per cell is $2 \cdot 476$. All crystals comprised several individuals giving rise to multiple spots, and the most suitable that could be found was one for which the separate reflexions coincided over half of the Weissenberg film, at least when the equi-inclination angle was very small. Intensities were measured visually from such spots for the layers $h k 0$ and $h k 1$. Spherical absorption corrections were applied.

This structure was also confirmed as previously described (Ketelaar \& von Walsem, 1938), i.e. as in Fm3m with palladium in (a), $0,0,0$; nitrogen in (c), $\frac{1}{4}, \frac{1}{4}, \frac{1}{4}$; and chlorine in (e), $x, 0,0$, with $x \sim 0 \cdot 23$. Refinement was then as before, except that only the chlorine could adopt anisotropic thermal parameters, and separate scale factors were used for the $h k 0$ and $h k 1$ data. Eight terms appeared to suffer from extinction and were removed. The $R$ index for the 29 terms included was $0 \cdot 071$. The final parameters are listed in Table 1 , and observed and calculated structure factors in Table 2.

The palladium-chlorine bond length is $2.300 \AA$, with $\sigma=0.007 \AA$.

The assumption that $\mathrm{Pd}(\mathrm{II})$ and $\mathrm{Pd}(\mathrm{IV})$ have effectively the same atomic radius has thus been confirmed.

We are indebted to the Research Committee of the New Zealand University Grants Committee for financial assistance.

## References

Berghuis, J., HaAnappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. \& Veenendaal, A. L. (1955). Acta Cryst. 8, 478.
Brosset, C. (1948). Ark. Kemi Min. Geol. A, 25, 1.
Cohen, A. \& Hughes, E. W. (1954). Structure Reports, 18, 538.

Craven, B. M. \& Hall, D. (1961). Acta Cryst. 14, 475.
Craven, B. M. \& Hall, D. (1966). Acta Cryst. 21, 177.
Dickinson, R. G. (1922). J. Amer. Chem. Soc. 44, 2404.
Hall, D. \& Williams, P. P. (1958). Acta Cryst. 11, 624.
Ketelaar, J. A. \& van Walsem, J. F. (1938). Rec. Trav. chim. Pays-Bas, 57, 964.
Main, P. \& Woolfson, M. M. (1963). Acta Cryst. 16, 731.
Pauling, L. (1960). The Nature of the Chemical Bond. 3rd Ed. Ithaca: Cornell Univ. Press.
Ryan, T. D. \& Rundle, R. E. (1961). J. Amer. Chem. Soc. 83, 2814.
Sharpe, A. G. (1953). J. Chem. Soc. p. 4177.
Thomas, L. A. \& Umeda, K. (1957). J. Chem. Phys. 26, 293.
Wallen, J., Brosset, C. \& Vannerberg, N. (1962). Ark. Kemi, 18, 541.

Acta Cryst. (1966). 21, 442
Further refinement of the crystal structure of acetanilide. By C. J. Brown, The Sir William Ramsay and Ralph
Forster Laboratories, University College, Gower Street, London, W.C.1, England
(Received 29 March 1966)

As a result of a number of requests the crystal structure of acetanilide (Brown \& Corbridge, 1954) has been further refined. The original X-ray intensity data were used and Cruickshank's (1961) program was used for refining the positional and anisotropic thermal parameters on a Pegasus computer. Seven cycles of structure factors and least squares reduced $R$ from $11.2 \%$ to $5.9 \%$ taken over the 1125 observed $F(h k l)$. The structure amplitude agreement is given in Table 1, the new atomic parameters in Table 2, and the bond lengths and inter-bond angles in Table 3.

The equation of the mean plane through the benzene ring is

$$
0.2049 X-0.5482 Y-0.8108 Z=0.4229
$$

from which the atoms are displaced by $\mathrm{C}(1)-0.014 ; \mathrm{C}(2)$ $+0.009 ; C(3)+0.001 ; C(4)-0.010 ; C(5) 0 ; C(6)+0.013 \AA$. The nitrogen atom is $-0.046 \AA$ out of this plane so that the $\mathrm{C}-\mathrm{N}$ bond makes an angle of $1.9^{\circ}$ with the plane of the ring.

The equation of the mean plane containing $C(7), C(8)$, N and O is

$$
0.3689 X-0.2990 Y-0.8801 Z=1.8978
$$

from which the atoms are displaced by $\mathrm{C}(7)+0.002 ; \mathrm{C}(8)$ $0 ; \mathrm{N}-0.001$; and $\mathrm{O}-0.001 \AA$. The normals to these two planes are inclined at $17.6^{\circ}$; the value of $37^{\circ} 54^{\prime}$ given in the previous paper was wrong.

The hydrogen bond, assumed to be linear, makes angles of $110.2^{\circ}$ with the $\mathrm{N}-\mathrm{C}(1)$ bond, $121.9^{\circ}$ with $\mathrm{N}-\mathrm{C}(7)$, and $139 \cdot 1^{\circ}$ with $\mathrm{O}^{\prime}-\mathrm{C}\left(7^{\prime}\right)$.

The results of this further refinement seem to indicate an improvement in overall regularity of the molecule; the benzene ring is more nearly regular and more planar; the acetyl group is now planar, and the mean standard deviation of a bond has been reduced from 0.0056 to $0.0034 \AA$. There is no change in the conformation of the molecule; the mean change in non-hydrogen positional coordinates is only $0.012 \AA$. This is of interest since in the structure of $N$ methylacetanilide (Pedersen \& Pedersen, 1965) the C(8) and O atoms have changed places, together with other differences in molecular geometry. Presumably this exo configuration is the more stable one when the oxygen atom does not have to be forced into the endo configuration to accept a hydrogen bond.

