and Noyes[3] have demonstrated a similar reaction. Extensions of these investigations are being continued.

(3) G. H. Coleman and W. A. Noyes, THIS JOURNAL, **43**, 2211 (1921).

CHEMICAL RESEARCH DEPT. M. E. BROOKS
DAVISON CHEMICAL COMPANY B. RUDNER
BALTIMORE 3, MARYLAND

RECEIVED APRIL 12, 1956

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## CRYSTAL STRUCTURE AND MAGNETIC SUSCEPTIBILITY OF AMERICIUM METAL[1]

*Sir:*

We have obtained interpretable X-ray diffraction patterns of several small polycrystalline samples of americium metal of $>99\%$ purity, using Cu K$\alpha$ radiation and a 4.5-cm. radius camera. The metal was prepared on a micro scale by reduction of the trifluoride with barium vapor, in a tantalum crucible system and subsequently was annealed by slowly reducing the temperature from 800 to *ca.* 25° over a period of ten hours.

The powder patterns have been indexed as double hexagonal close packed, $a = 3.642 \pm 0.005$ Å., $c = 11.76 \pm 0.01$ Å.

The space group is $D_{6h}^4$ and the atomic positions are: two Am in (0, 0, 0), (0, 0, $^1/_2$); two Am in ($^1/_3$, $^2/_3$, $^3/_4$), ($^2/_3$, $^1/_3$, $^1/_4$). The Am radius is 1.82 Å. and the calculated density $11.87 \pm 0.05$ g. cm.$^{-3}$.

Relative line intensities calculated for the proposed structure agreed with visual estimates of the intensities seen in the diffraction patterns, as shown in the accompanying table.

The density calculated for the metal agrees with that observed experimentally[2] ($11.7 \pm 0.3$) within the error of the measurements. The metallic radius is 0.02 Å. smaller than that predicted by Zachariasen[3] for americium metal with three valence electrons per atom. This discrepancy may indicate a small error in the predicted value, or may be due to a slight admixture of americium (IV) in the metallic state. Measurements of the magnetic susceptibility of our samples gave $\chi_M = 1000 \pm 250 \times 10^{-6}$ cgs. units at 300° K., similar to the value of $\sim 1000 \times 10^{-6}$ c.g.s. units for AmF$_3$. The number of bonding electrons per atom appears to be quite close to three.

The decrease in the number of metallic bonds in going from uranium to americium affords a reasonable explanation of the corresponding decrease of some 50 kcal.[4,5] in the heat of vaporization.

It is interesting to note that americium is the first transactinium element which is rare earth-like in the metallic state.

Possible allotropy of the metal is now under investigation, and these studies, as well as a detailed description of the work outlined above, will be reported in a future publication.

TABLE I

DIFFRACTION DATA FOR AMERICIUM METAL[a]

| *hkl* | $\sin^2\theta$ calcd. | $\sin^2\theta$ obs. | *I* calcd. | *I* obs.[b] |
|---|---|---|---|---|
| 100 | 0.0597 | 0.0592 | 3 | vw |
| 101 | .0640 | .0640 | 18 | m |
| 004 | .0687 | .0692 | 14 | ms |
| 102 | .0769 | .0769 | 42 | s |
| 103 | .0984 | .0985 | 10 | w |
| 104 | .1284 | .1283 | 2 | t |
| 105 | .1670 | .1675 | 5 | vw |
| 110 | .1792 | .1792 | 11 | m |
| 106 | .2142 | .2144 | 9 | m |
| 200 | .2389 | .... | 0.4 | .. |
| 201 | .2432 | .... | 3 | .. |
| 114 | .2479 | .2471 | 13 | ms |
| 202 | .2561 | .2567 | 7 | vw |
| 107 | .2700 | .2698 | 2 | t |
| 008 | .2747 | .2755 | 2 | vvw |
| 203 | .2776 | | 2 | |
| 204 | .3076 | .... | 0.6 | .. |
| 108 | .3344 | .... | 0.5 | .. |
| 205 | .3462 | .3462 | 1 | t |
| 206 | .3934 | .3930 | 4 | vvw |
| 109 | .4074 | .4070 | 1 | t |
| 210 | .4181 | .... | 0.4 | .. |
| 211 | .4224 | .4226 | 2 | t |
| 212 | .4353 | .4353 | 0.6 | t |
| 207 | .4492 | .... | 1 | .. |
| 118 | .4539 | .4531 | 5 | m |
| 213 | .4567 | | 2 | |
| 214 | .4867 | .4878 | 0.6 | vw |
| 1,0,10 | .4889 | | 2.6 | |

[a] This list includes all planes up to $\sin^2\theta = 0.5$ for which the intensity was not calculated to be zero, by the symmetry of the special positions. [b] t, trace; vvw, very, very weak; vw, very weak; w, weak; m, moderate; ms, moderately strong; s, strong.

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PETER GRAF
DEPARTMENT OF CHEMISTRY AND B. B. CUNNINGHAM
RADIATION LABORATORY CAROL H. DAUBEN
UNIVERSITY OF CALIFORNIA J. C. WALLMANN
BERKELEY, CALIFORNIA D. H. TEMPLETON
HELENA RUBEN

RECEIVED FEBRUARY 27, 1956

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(1) This work was performed under the auspices of the AEC.

(2) E. F. Westrum, Jr., and L. Eyring, THIS JOURNAL, **73**, 3396 (1951).

(3) W. H. Zachariasen, *Acta Cryst.*, **5**, 660 (1952).

(4) E. G. Rauh and R. J. Thorn, *J. Chem. Phys.*, **22**, 1414 (1954).

(5) S. C. Carniglia and B. B. Cunningham, THIS JOURNAL, **77**, 1502 (1955).

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## SYNTHESIS OF FUSED-RING COMPOUNDS *VIA* ACYLATION OF KETONES

*Sir:*

A new method for synthesis of fused-ring compounds (II) has been found, which offers certain advantages over the well-known Pschorr,[1] Haworth,[2] Bardhan–SenGupta[3] and Bogert[4] procedures. The method consists of the reaction of cyclic ketones with phenylacetic anhydrides in the presence of boron trifluoride.[5] Acylation and cy-

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(1) R. Pschorr, *Ber.*, **29**, 496 (1896).

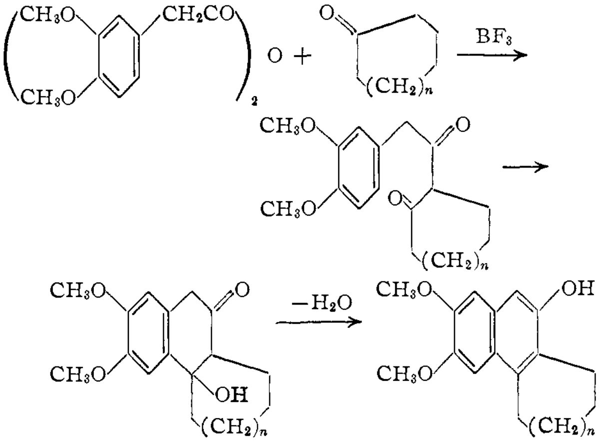
(2) R. D. Haworth, *J. Chem. Soc.*, 1125 (1932).

(3) J. C. Bardhan and S. C. SenGupta, *ibid.*, 2520 (1932).

(4) M. T. Bogert, *Science*, **77**, 289 (1933).

(5) C. R. Hauser, F. W. Swamer and J. T. Adams, in R. Adams, "Organic Reactions," Vol. 8, Chapter 3, pp. 59–196, J. Wiley and Sons, Inc., New York, N. Y., 1954.

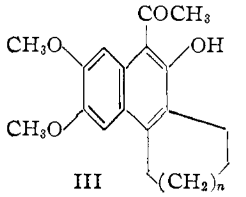
clization are accomplished together in one step and substituted β-naphthols or 4-hydroxy-2-tetralones are isolated.

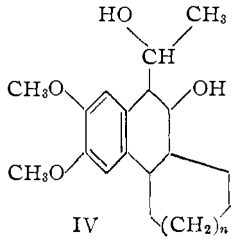


I II

A mixture of homoveratric anhydride,[6] cyclohexanone and boron trifluoride etherate was allowed to stand for three days, and the boron complex was decomposed with sodium acetate solution. Compound I ($n$ = 1), m.p. 140–141° (*Anal.* Calcd. for $C_{16}H_{20}O_4$: C, 69.54; H, 7.30. Found: C, 69.55; H, 7.34), showing infrared absorption at 2.8–2.9 and 5.79–5.85 μ, was isolated in 68% yield. Dehydration by brief treatment with polyphosphoric acid gave II ($n$ = 1), m.p. 219–221° (*Anal.* Calcd. for $C_{16}H_{18}O_3$: C, 74.40; H, 7.02. Found: C, 74.50; H, 7.09). Similar condensation of homoveratric anhydride with cycloheptanone gave II ($n$ = 2) directly (29%), m.p. 207–208° (*Anal.* Calcd. for $C_{17}H_{20}O_3$: C, 74.97; H, 7.40. Found: C, 74.93; H, 7.45). The infrared spectra of compounds II each showed a band at 2.76 μ. The ultraviolet spectra of II ($n$ = 1) ($\lambda_{\max}^{\text{EtOH}}$ 237, 272, 282, 293, 322 and 336 mμ; log ε 4.86, 3.60, 3.59, 3.46, 3.59 and 3.72, respectively) and of II ($n$ = 2) ($\lambda_{\max}^{\text{EtOH}}$ 237, 274, 285, 296, 324 and 337 mμ; log ε 4.91, 3.61, 3.61, 3.49, 3.58, and 3.72, respectively) were typical of naphthalene derivatives. O-Acetates of II ($n$ = 1), m.p. 170–171° (*Anal.* Calcd. for $C_{18}H_{20}O_4$: C, 71.98; H, 6.71. Found: C, 72.03; H, 6.69) and of II ($n$ = 2), m.p. 139–141° (*Anal.* Calcd. for $C_{19}H_{22}O_4$: C, 72.59; H, 7.06. Found: C, 72.84; H, 7.06) had similar ultraviolet spectra and absorbed light at 5.70 μ in the infrared region.

When boron trifluoride in acetic acid was used in condensation of homoveratric anhydride with cyclohexanone and cycloheptanone, acetylation of the phenolic ring also occurred, probably through Fries rearrangement of O-acetates, giving III ($n$ = 1), m.p. 167–168° (*Anal.* Found: C, 72.01; H, 6.75) and III ($n$ = 2), m.p. 157–158° (*Anal.* Found: C, 72.64; H, 7.06) in yields of 38% and 11%, respectively. These yellow compounds gave characteristic ferric chloride tests and had infrared spectra similar to that of 1-hydroxy-2-acetonaphthone, with no hydroxyl or normal carbonyl peaks. The ultraviolet spectra of III ($n$ = 1) ($\lambda_{\max}^{\text{EtOH}}$ 234, 294 and 340, mμ; log ε 4.80, 3.57 and 4.00, respectively) and of III ($n$ = 2) ($\lambda_{\max}^{\text{EtOH}}$ 235, 291 and 345 mμ; log ε 4.84, 3.56 and 4.02, respectively) again were typical of naphthalene derivatives. Trihydrogenation of hydroxy ketones III ($n$ = 1 and 2) in the presence of palladium-charcoal in ethyl acetate at 80° gave diols IV ($n$ = 1), m.p. 171–173° (*Anal.* Calcd. for $C_{18}H_{26}O_4$: C, 70.56; H, 8.55. Found: C, 70.50; H, 8.23) and IV ($n$ = 2), m.p. 142–144° (*Anal.* Calcd. for $C_{19}H_{28}O_4$: C, 71.22; H, 8.81. Found: C, 71.44; H, 8.75), respectively. The infrared spectra of compounds IV ($n$ = 1 and 2) each had a doublet at 2.76–2.98 μ, and their ultraviolet spectra had $\lambda_{\max}^{\text{EtOH}}$ 288 mμ (log ε 3.34) and $\lambda_{\max}^{\text{EtOH}}$ 291 mμ (log ε 3.43), respectively.


III


IV

The synthesis permits variation of the size of ring C and thus will be useful in getting compounds related to colchicine[7] as well as new phenanthrenes.

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[6] Prepared by refluxing homoveratric acid with acetic anhydride in the presence of pyridine; m.p. 83–84°.

[7] H. Rapoport, *et al.*, THIS JOURNAL, **73**, 1414 (1951); **77**, 670 (1955).

LABORATORY OF CHEMISTRY OF NATURAL PRODUCTS
NATIONAL HEART INSTITUTE
NATIONAL INSTITUTES OF HEALTH GORDON N. WALKER
DEPARTMENT OF HEALTH, EDUCATION AND WELFARE
BETHESDA 14, MARYLAND

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## THE CONFIGURATION OF (+)-α-LIPOIC ACID

*Sir:*

The synthesis of naturally occurring (+)-α-lipoic acid[1] has recently been described.[2] We wish to report on the elucidation of its stereochemistry.

(+)-3-Acetylthio-7-carbethoxyheptanoic acid (Ia)[2,3] was treated with cold 10% sodium hydroxide to give 3-thioloctanedioic acid (IIa), m.p. 114.5–115.5°, $[\alpha]^{27}_{D}$ −7.9° ($c$ 3.4, pyridine); calcd. for $C_8H_{14}O_4S$: C, 46.6; H, 6.8; S, 15.6; found: C, 46.6; H, 6.6; S, 15.4. Similarly (−)-3-acetylthio-7-carbethoxyheptanoic acid (Ib)[2,3] gave 3-thioloctanedioic acid (IIb), m.p. 115–116°, $[\alpha]^{27}_{D}$ +7.9° ($c$ 2.5, pyridine), calcd. for $C_8H_{14}O_4S$: C, 46.6; H, 6.8; S, 15.6; found, C, 46.8; H, 6.8; S, 15.4.

Benzyl hydrogen glutarate was prepared from glutaric anhydride, benzyl alcohol and pyridine, b.p. 153–155° (0.05 mm.), $n^{25}_{D}$ 1.5108; calcd. for $C_{12}H_{14}O_4$: C, 64.8; H, 6.4; neut. eq., 222; found: C, 65.0; H, 6.1; neut. eq., 221. Anodic cross-coupling with (+)-methyl hydrogen β-methyl-

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[1] L. J. Reed, I. C. Gunsalus, G. H. F. Schnakenberg, Q. F. Soper, H. E. Boaz, S. F. Kern and T. V. Parke, THIS JOURNAL, **75**, 1267 (1953); E. L. Patterson, J. V. Pierce, E. L. R. Stokstad, C. E. Hoffmann, J. A. Brockman, Jr., F. P. Day, M. E. Macchi and T. H. Jukes, *ibid.*, **76**, 1823 (1954).

[2] E. Walton, A. F. Wagner, F. W. Bachelor, L. H. Peterson, F. W. Holly and K. Folkers, *ibid.*, **77**, 5144 (1955).

[3] A sample was kindly supplied by Dr. F. M. Robinson, Merck and Co.