

The final structure for the  $X_2O_2S$  compounds is:

Space group:  $C\bar{3}m-D_{3d}^3$ .

Atomic positions:

2 X in  $\pm(\frac{1}{3}, \frac{2}{3}, u_1)$ , with  $u_1 = 0.29 \pm 0.02$ ;

1 S in (0, 0, 0);

2 O in  $\pm(\frac{1}{3}, \frac{2}{3}, u_2)$ , with  $u_2 = 0.64$ .

The intensities calculated for  $Ce_2O_2S$  on the basis of this structure are given in the last column of Table 2. The intensity formula

$$I \propto |F|^2 \rho \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta}$$

was used. It is seen that reflections  $H_1H_2H_3$  with large values of  $H_3^2/(H_1^2 + H_2^2 + H_3^2)$  are observed with enhanced intensity. This is probably due to the crystallites being plates normal to the six-fold axis and to preferential orientation of the plates parallel to the surface of the briquet specimen.

### Discussion of the structure

The  $Ce_2O_2S$  structure is closely related to that of  $Ce_2O_3$  (type  $D52$ ), as the following comparison shows:

	$Ce_2O_2S$	$Ce_2O_3$
Space group	$C\bar{3}m$	$C\bar{3}m$
$a_1$	4.00 kX.	3.88 kX.
$a_3$	6.82 kX.	6.06 kX.
2Ce in	$\pm(\frac{1}{3}, \frac{2}{3}, u_1)$	$\pm(\frac{1}{3}, \frac{2}{3}, u_1)$
$u_1$	0.29	0.235
1S or 1O in	(0, 0, 0)	(0, 0, 0)
2O in	$\pm(\frac{1}{3}, \frac{2}{3}, u_2)$	$\pm(\frac{1}{3}, \frac{2}{3}, u_2)$
$u_2$	0.64	0.63

The  $Ce_2O_2S$  structure is in other words obtained from the  $Ce_2O_3$  structure by substituting a sulfur atom for

one of the three oxygen atoms in each unit cell and by appropriately distorting the resulting structure.

Each metal atom is bonded to four oxygen atoms and to three sulfur atoms. Table 3 shows the observed interatomic distances as well as those calculated from ionic radii (Zachariasen, 1931, 1948). It is shown that the observed X-O distances are somewhat smaller, the X-S distances somewhat larger than the values to be expected from the ionic radii. A shift of the parameter value  $u_1$  from 0.29 to 0.26 would remove this apparent discrepancy. However, the intensity considerations rule out a  $u_1$  value as low as 0.26. The reduction in the X-O distances and the increase in the X-S distances relative to the expected values thus seem to be real. It is not obvious why the X-O bonds are strengthened at the expense of the X-S bonds.

Other lanthanide elements as well as actinium and americium may be expected to form oxy-sulfides isostructural with  $Ce_2O_2S$ . Oxy-sulfides of thorium, uranium and neptunium are known; but these are of the form XOS and have the PbFCl type of structure.

The writer thanks Miss Anne Plettinger for having taken the X-ray diffraction patterns, Prof. L. Brewer for the various cerium oxy-sulfide samples and Dr N. R. Davidson for the loan of his micro-preparation of plutonium sulfide.

### References

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Table 3. *Interatomic distances*

	Observed			Calculated from ionic radii	
	X-1O (A.)	X-3O (A.)	X-3S (A.)	X-4O (A.)	X-3S (A.)
$X_2O_2S$					
$La_2O_2S$	2.41	2.38	3.06	2.44	2.92
$Ce_2O_2S$	2.39	2.36	3.04	2.42	2.90
$Pu_2O_2S$	2.37	2.33	2.99	2.41	2.89

## Short Communications

*Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible, and proofs will not generally be submitted to authors. Rapid publication will be easier if the contributions are without illustrations.*

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**The constitution of the dimer of acenaphthylene.** By J. D. DUNITZ and L. WEISSMAN, *Laboratory of Chemical Crystallography, University Museum, University of Oxford, England*

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An X-ray crystal analysis of the high-melting form of the dimer,  $C_{28}H_{24}$ , obtained by irradiation of stilbene,  $C_{14}H_{12}$ , has shown it to be the centrosymmetric isomer of 1:2:3:4-tetraphenylcyclobutane (Fulton & Dunitz, 1947).

Further analysis has revealed the molecular structure, and furnishes strong evidence that the C-C distance in the central ring is greater than 1.54 Å., the normal single-bonded C-C distance (Dunitz, 1948).

The corresponding dimer,  $C_{24}H_{16}$ , of acenaphthylene,  $C_{12}H_8$ , (I) has now been examined. The X-ray data for the two isomers obtained by irradiation of acenaphthylene (Dziewonski & Rapalski, 1912) are as follows:

Melting point ( $^{\circ}$ C.)	307	234
Crystal system	Monoclinic	Monoclinic
$a$ (A.)	$7.81 \pm 0.02$	$9.91 \pm 0.05$
$b$ (A.)	$4.86 \pm 0.01$	$13.81 \pm 0.05$
$c$ (A.)	$20.16 \pm 0.05$	$12.14 \pm 0.06$
$\beta$ ( $^{\circ}$ )	$92 \pm 0.5$	$106 \pm 1.0$
Space group	$P2_1/n$	$P2_1/n$
Density at $18^{\circ}$ C. (g.cm. $^{-3}$ )	1.31	1.25

The observed densities are consistent with the unit cells containing two and four molecules of dimer respectively, and it follows that the high-melting crystals

plane, with the condensed planar systems of five- and six-membered rings inclined at about  $45^{\circ}$  to (010). The sides of the four-membered ring, even in projection, appear to be somewhat longer than 1.54 A., but accurate values are not yet available. Certain distortions from

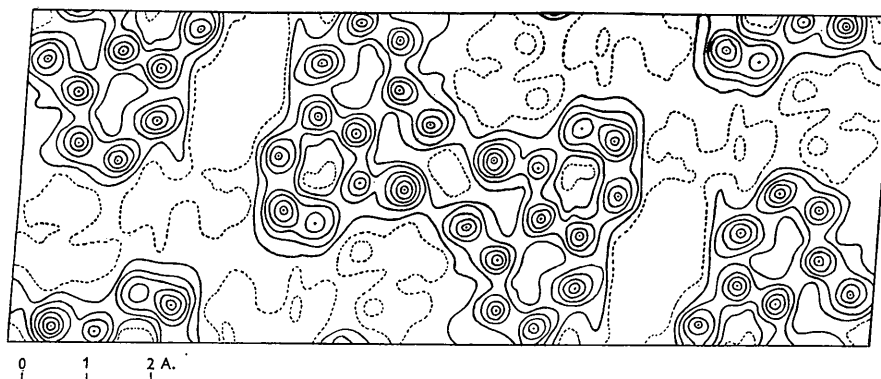
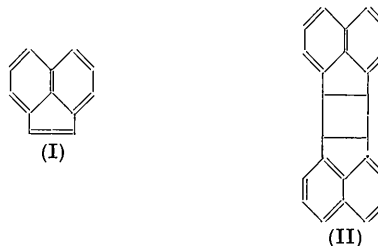


Fig. 1. Acenaphthylene dimer: Fourier projection on (010), showing one unit cell.

contain centrosymmetric molecules of the dimer. No molecular symmetry is imposed crystallographically on the low-melting isomer.

The structure determination of the centrosymmetric isomer has been commenced. Trial-and-error methods have been employed to fix the signs of the ( $h0l$ ) reflexions and a Fourier projection on (010) has been calculated and is shown in Fig. 1. There is no doubt that the molecule is dinaphthylene cyclobutane (II), in confirmation of the conclusions, based on chemical evidence, of Dziewonski & Rapalski (1912).

The atomic positions cannot yet be fixed with any precision, but certain features of the structure seem clear. The four-membered ring must lie almost in the (010)

regularity also appear to exist in the naphthylene groups.

Work is being continued with a view to obtaining more accurate values for the dimensions of the molecule.

We wish to thank Mrs D. Hodgkin (Dorothy Crowfoot) for her continued interest.

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**A preliminary X-ray investigation of some pyridindene derivatives.** By C. H. CARLISLE and S. FURBERG.  
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2-Methyl-9-phenyl-2, 3, 4, 9-tetrahydro-1-pyridindene (I), or thephorin (T.M. Reg. U.S. Pat. Off.), is reported to be a useful drug in hay fever and other allergic disorders. Closely related compounds (e.g. II), apparently only differing from thephorin in the position of a double bond, show very low biological activity. Samples of these interesting compounds were kindly sent us from the research laboratories of Hoffmann La Roche, Inc., Nutley,

N.J., where they were synthesized by Dr J. Plati and Dr W. Wenner.

We have carried out a preliminary X-ray investigation of four of the substances. They are: (1) thephorin (I), (2) the thiocyanate salt of thephorin, (3) 4a-9a-dihydroxy-dihydro-thephorin, and (4) the nitrate salt of a base, which is isomeric with thephorin and probably has formula (II).