2·490 Å, the corresponding Pt—Ge distances are 2·520, 2·563 and 2·577 Å. In Model 2, the Pt—Se distances are 2·526, 2·563 and 2·573 Å, those of Pt—Ge are 2·452, 2·455 and 2·492 Å. Hence, for either model, the minimum difference between Pt—Se and Pt—Ge interatomic distances is about 0·03 Å with a maximum range of about 0·06 Å in each of the two types of contact, suggestive of near equality between the two bond types. The atomic radii in PtGeSe, based on the average interatomic distances, are Pt 1·275, Ge 1·283, Se 1·191 Å for Model 1 and Pt 1·285, Ge 1·181, Se 1·269 Å for Model 2. Phillips's (1973) rationalized tetrahedral radii for Ge and Se are equal, at 1·225 Å, in contrast to Pauling's (1960) values of 1·22 Å for Ge and 1·14 Å for Se which were initially among EP's criteria (Parthé, 1976) in solving the crystal structure.

The ease with which large atomic displacements are produced in PtGeSe on ferroelastic reorientation together with the low resistivity at room temperature led to the suggestion by ABB that the bonding in PtGeSe tends toward metallic rather than pure covalent. Present bond theory appears unable, in this case, to distinguish between Pt—Se and Pt—Ge interatomic separations.

Experimentally, the Pt—Ge distance has been reported as ranging from 2·480 (6) to 2·656 (5) Å in PtGe by Graeber, Baughman & Morosin (1973), 2·433 (2) Å in *cis*-(hydroxydiphenylgermanyl)phenylbis(triethylphosphine)platinum(II) by Gee & Powell (1971), and ranging from 2·391 (3) to 2·480 (3) Å in tetramethylammonium pentakis(trichlorogermanyl)platinate(II) by Estes & Hodgson (1973). Heinrich & Schubert (1976) report coordinates for Pt_3Ge_2 that give a range of Pt—Ge distances from 2·535 to 2·690 Å. The only reliable Pt—Se distance found in the literature is the value 2·513 (9) Å reported for $PtSe_2$ by Furuseth, Selte & Kjekshus (1965). The interatomic distances cited for Pt—Se and Pt—Ge are consistent with the values that correspond to Model 2 for PtGeSe. It may be noted, as pointed out by Parthé (1976), that an attempted distinction between Ge and Se based on neutron scattering will be subject to ambiguities that are similar to those found in the X-ray study, since the neutron scattering amplitudes are $0{\cdot}819 \times 10^{-12}$ cm for Ge and $0{\cdot}80 \times 10^{-12}$ cm for Se (Bacon, 1972).

It is a pleasure to acknowledge discussions with E. Parthé and J. C. Phillips.

References

Abrahams, S. C., Bernstein, J. L. & Buehler, E. (1976). *Mater. Res. Bull.* **11**, 707–712.

Abrahams, S. C. & Reddy, J. M. (1965). *J. Chem. Phys.* **43**, 2533–2543.

Bacon, G. E. (1972). *Acta Cryst.* **A28**, 357–358.

Entner, P. & Parthé, E. (1973). *Acta Cryst.* **B29**, 1557–1560.

Estes, E. D. & Hodgson, D. J. (1973). *Inorg. Chem.* **12**, 2932–2935.

Furuseth, S., Selte, K. & Kjekshus, A. (1965). *Acta Chem. Scand.* **19**, 257–258.

Gee, R. J. D. & Powell, H. M. (1971). *J. Chem. Soc.* (*A*), pp. 1956–1960.

Graeber, E. J., Baughman, R. J. & Morosin, B. (1973). *Acta Cryst.* **B29**, 1991–1994.

Heinrich, S. & Schubert, K. (1976). *J. Less-Common Met.* **45**, 125–135.

International Tables for X-ray Crystallography (1974). Vol. IV, edited by J. S. Ibers & W. C. Hamilton. Birmingham: Kynoch Press.

Parthé, E. (1976). Private communication.

Pauling, L. (1960). *The Nature of the Chemical Bond.* Ithaca: Cornell Univ. Press.

Phillips, J. C. (1973). *Bonds and Bands in Semiconductors.* New York: Academic Press.

Acta Cryst. (1977). **B33**, 302–303

The absolute configuration of apomorphine.

By J. Giesecke, *Department of Medical Biophysics, Karolinska Institutet, S-104 01 Stockholm, Sweden*

(*Received* 24 *May* 1976; *accepted* 10 *June* 1976)

The absolute configuration of apomorphine was determined with anomalous scattering. 23 Friedel pairs were recorded on film. As has previously been shown with chemical methods it was found that the absolute configuration at the only asymmetric carbon atom is *R*.

Apomorphine is a very important substance in the study of humoural neurotransmission as it acts directly on dopamine receptors. From an inspection of a model of the apomorphine molecule it is immediately evident that the proton of the charged N atom can only be approached from one side of the rather planar molecule. Since this proton presumably plays an important role in the neurotransmission, knowledge of the absolute configuration of apomorphine would indirectly bring information not only about the receptor, but also about the optimum conformation of related neurotransmitters.

Using chemical methods, Kalvoda, Buchschacher & Jeger (1955) assumed, and Corrodi & Hardegger (1955) confirmed that the configuration of apomorphine is *R* at the only asymmetric carbon atom (6a). Since a suitable crystal was left from a previous investigation (Giesecke, 1973) it was interesting to make a redetermination of the absolute configuration by X-ray methods.

Experimental

A crystal of apomorphine hydrobromide, roughly cylindrical about the *c* axis, was mounted about **c** in a Nonius Weissenberg camera. Zr-filtered Mo $K\alpha$ radiation was chosen since this wavelength gives a favourable anomalous scattering factor for bromine (*International Tables for X-ray Crystallography*, 1974). The layer *hk*1 was recorded by the integrated Weissenberg technique. The exposure time was 120 h.

Results

For the space group $P2_12_12_1$, $F(hkl)=F(\bar{h}\bar{k}\bar{l})=F(\bar{h}kl)=F(h\bar{k}l)$ for $l>0$, and thus the Friedel pairs are symmetrically distributed about the *h*01 and 0*k*1 axial rows. To find the pairs exhibiting the greatest anomalous dispersion effect the structure factor amplitudes of all *hk*1 with $|h|\leq 10$, $|k|\leq 10$ were calculated with the coordinates determined earlier. The members of 23 pairs differed by more than 10% in their calculated amplitudes. The reflexions of these pairs were measured on a microdensitometer, and 15 pairs were intense enough to give satisfactory readings. $F_{\rm calc}$ for these 30 reflexions were then calculated from the absolute configuration which is *R* at the C atom designated C(8) by Giesecke (1973), 6a in chemical literature. It was found that for all 15 pairs whenever $F^+_{\rm obs} > F^-_{\rm obs}$ then $F^+_{\rm calc} > F^-_{\rm calc}$ and *vice versa*.

The conclusion is that the correct absolute configuration of apomorphine is *R*. The arbitrarily chosen enantiomorph presented in the structure determination of apomorphine (Giesecke, 1973) is thus incorrect.

References

Corrodi, H. & Hardegger, E. (1955). *Helv. Chim. Acta*, **38**, 2038–2043.

Giesecke, J. (1973). *Acta Cryst.* **B29**, 1785–1791.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 149. Birmingham: Kynoch Press.

Kalvoda, J., Buchschacher, P. & Jeger, O. (1955). *Helv. Chim. Acta*, **38**, 1847.

Acta Cryst. (1977). B**33**, 303–305

Molecular configuration of a tridecazirconium oxide–methoxide complex.*

By B. Morosin, *Sandia Laboratories, Albuquerque, New Mexico* 87115, *USA*

(*Received* 31 *March* 1976; *accepted* 23 *June* 1976)

Molecules of $Zr_{13}O_8(OCH_3)_{36}$ are found as a hydrolysis product in very unstable crystals [$R\,\bar{3}m$, $a=17{\cdot}31$ (1), $c=27{\cdot}75$ (2) Å]; the cubic close-packed arrangement of Zr (three above and below a plane of seven) consists of a central Zr ion bonded to eight oxide ions and 12 sevenfold-coordinate Zr ions each bonded to two oxide ions, four bridging methoxide groups and one terminal methoxide group.

Our laboratories have used the alkoxides of Ti, Nb and Zr as starting materials for the preparation of the perovskite-class of ferroelectrics and electrooptic ceramics (Dosch, 1971, 1972; Haertling & Land, 1972) and, more recently, ion-exchange materials which can be used to stabilize radioactive wastes (Lynch, Dosch, Kenna, Johnstone & Nowak, 1976). Most such precipitated materials yield amorphous diffraction patterns (Morosin & Peercy, 1976); however, crystals of a rather unstable polyzirconium oxide–methoxide intermediate were grown. The only structural study on such a metal oxide–alkoxide complex, or first hydrolysis product, which has been reported appears to be that for Ti (Watenpaugh & Caughlan, 1967), although structures of the alkoxides of Ti (Ibers, 1963; Witters & Caughlan, 1965) and Tl (Dahl, Davis, Wampler & West, 1962) are known.

Very unstable crystals containing molecules of composition $Zr_{13}O_8(OCH_3)_{36}$ plus disordered entities (possibly OH^-, Na^+ and CH_3OH) were obtained on the walls of bottles containing solutions made by mixing dilute methanol solutions of NaOH and the alkoxide of zirconium (prepared and kindly supplied by R. G. Dosch). The crystals are very sensitive to water as well as unstable in dry air. Under crossed polars in dried methyl alcohol, one can observe such crystals undergoing a spontaneous transformation (instantaneous over the entire crystal) into an amorphous material, presumably as atmospheric water diffuses through the alcohol into the crystal. Chemical analysis of small amounts of such an amorphous product showed the presence of ~3 wt% Na, with the ashed residue (ZrO_2) determined to be ~ 64%. A crudely determined density of $1{\cdot}78$ g cm^{-3} is consistent with $Z=3$ molecules of $Zr_{13}O_8(OCH_3)_{36}$.

Diffraction data were obtained on various specimens encapsulated in glass capillaries filled with oil. All the specimens showed some radiation degradation. A set of intensities (2037 independent reflections to $2\theta=55°$ with Mo $K\alpha$) was obtained by normalizing against a rapidly collected (3° min^{-1}) small set, the radiation-corrected (using standards) θ–2θ scanned (1° min^{-1}, 20 s background count times) data sets. No attempt was made to correct for the absorption of the oil or glass capillary. A Patterson synthesis yielded the Zr positions, and subsequent Fourier synthesis together with least-squares refinement of positional and thermal parameters yielded the molecular structure of the tridecazirconium oxide–methoxide complex (Fig. 1). The func-

---

* Work supported by the US Energy Research and Development Administration.