minating structons of the G′ type. The ions cannot be infinite in size, nor can they be rings—assuming like atoms to be bonded in like manner. For reasons that have been discussed, compositions with mixtures of G′ and G″ structons do not readily crystallize (especially from the molten state), unless the relative proportions of these two types are such as to lead to only a single ionic composition. Sodium triphosphate is the only example, of known crystal structure, that the author has been able to find.

With σ equal to 3.5 and 4, the G structons are of the G′ and G^0 types, respectively. The anions are small and of the expected compositions and structures.

Conclusion

It has been shown that the structon concept, when applied in connection with other well-established structural principles, leads to conclusions in agreement with a large body of crystal structure knowledge. One can now confidently apply this concept and the procedures here outlined to crystals outside of the class included in the present treatment. One can also use them confidently to predict characteristics of the structures of crystals and glasses that have not yet been successfully analyzed experimentally.

Knowing the types of structons and the numbers of each type for many substances, one can now proceed to use this knowledge to compute the magnitudes of various properties by adding together the contributions to those properties of the different types of structons. It is predicted that considerably greater accuracy will be achieved in this way, for many properties, than by addition of the contributions of the individual atoms.

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The Fluorination of Metal Stannates[1]

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The reaction of fluorine at about 500° with a series of anhydrous metal stannates has been studied. The resultant anhydrous fluorostannates are $MgSnF_6$, $CaSnF_6$, $SrSnF_6$, $BaSnF_6$, $CoSnF_6$, $NiSnF_6$, $CuSnF_6$, $ZnSnF_6$, $CdSnF_6$, $MnSnF_8$, and $PbSnF_8$. The hexafluorostannates contain the SnF_6^{2-} anion, and X-ray diffraction and infrared spectroscopy have been used to postulate structures for some of these compounds.

Introduction

Studies of anhydrous fluorometalates of the general formula A_xBF_y have usually dealt with complexes in which A is an alkali metal or an ammonium ion, although a few alkaline earth, silver, and thallium complexes have also been studied. Babel[3] has recently reviewed the structural chemistry of octahedral fluoro complexes of the transition elements, and Wells[4] has discussed the general structural relationships.

Direct replacement of oxygen by fluorine at temperatures above 500° often proceeds smoothly and completely. The availability of many ternary oxides, A_mBO_n, offers a potential starting point for syntheses of anhydrous fluorometalates in which the type of A atom can vary widely. Many metal stannates can be prepared from readily available commercial products, and we have studied the complexes produced by direct fluorination of 11 of these. Formation of such anhydrous fluorostannates from aqueous solution or by direct reaction of tin(IV) fluoride and metal fluoride is not practical.

Experimental Section

Fluorination.—Samples of hydrated metal stannates, $MSnO_3 \cdot 3H_2O$,[5] were dehydrated at appropriate temperatures, as indicated by earlier thermal decomposition studies.[6] These temperatures are shown in Table I. Fluorine was generated in a Harshaw laboratory-type fluorine cell at an approximate rate of 2 l./hr and was passed through a Dry Ice and trichloroethylene trap and sodium fluoride scrubber at 100° to remove residual hydrogen fluoride. The fluorine was then passed into a heated nickel reaction chamber containing a nickel boat with the dehydrated stannate. Residual fluorine was destroyed in a soda lime scrubber.

Fluorine was first introduced at room temperature, and the sample temperature was then increased to 500° over a 1-hr period. The sample was cooled in fluorine, the fluorine was purged with nitrogen, and the product was removed, pulverized, and then refluorinated for about 30 min at 500°. Fluorination was repeated three or four times. The products were subsequently handled in a nitrogen atmosphere.

Analysis.—Fluorine was determined by oscillometric titration[7] after distillation in the Tenso-Lab apparatus.[8]

Tin was determined by atomic absorption spectrometry, using a Techtron AA-3 unit, with the tin resonance line at 2863 Å.[9] A tin concentration range of 200–1000 ppm was used for all analyses. Standards and samples were dissolved in 0.5 *M* hydro-

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[1] Taken in part from the Ph.D. thesis of P. J. Moehs. Supported by the U. S. Atomic Energy Commission.

[2] To whom communications should be addressed.

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TABLE I

PROPERTIES OF THE FLUOROSTANNATES

Dehydration temp of stannate, °C	Fluorostannate formed	Color	Element determined	% calcd	% found	Magnetic moment, μ_{eff}, BM
750	$MgSnF_6$	White	Mg	9.46	8.78	0.0
			Sn	46.19	46.15	
			F	44.35	44.06	
700	$CaSnF_6$	White	Ca	14.69	14.81	0.0
			Sn	43.52	43.49	
			F	41.79	41.63	
500	$SrSnF_6$	White	Sr	27.36	27.14	0.0
			Sn	37.06	37.37	
			F	35.59	35.10	
650	$BaSnF_6$	White	Ba	37.12	37.12	0.0
			Sn	32.08	32.57	
			F	30.81	30.33	
950	$CoSnF_6$	White	Co	20.01	20.13	5.12
			Sn	40.70	40.90	
			F	39.52	39.12	
220	$NiSnF_6$	Yellow	Ni	20.14	20.84	3.40
			Sn	40.74	40.94	
			F	39.12	39.05	
320	$CuSnF_6$	White	Cu	21.45	20.81	2.01
			Sn	40.07	39.77	
			F	38.48	38.17	
350	$ZnSnF_6$	White	Zn	21.93	21.88	0.0
			Sn	39.82	39.49	
			F	38.24	37.89	
700	$CdSnF_6$	White	Cd	33.04	33.06	0.0
			Sn	34.40	34.27	
			F	33.04	33.04	
300	$MnSnF_8$	Olive	Mn	16.87	17.05	4.62
			Sn	36.45	35.81	
			F	46.68	46.70	
200	$PbSnF_8$	White	Pb	43.46	42.93	0.0
			Sn	24.84	23.06	
			F	31.80	30.84	

chloric acid containing 10–20 drops of 48% hydrofluoric acid per 250 ml of solution. These standards were more stable than those made with hydrochloric acid alone. Tin was determined as oxide in the cadmium, zinc, and lead complexes.

Magnesium, cobalt, nickel, copper, and manganese were also determined by atomic absorption spectrometry.[9] Calcium was determined by precipitation as oxalate after removal of tin by electrolysis.[10] Strontium, barium, and lead were precipitated as sulfates; zinc was determined as zinc ammonium phosphate.

X-Ray Diffraction.—Powder diffraction patterns were taken with a 114.6-mm Philips camera. Ground samples were mounted in 0.3-mm glass capillaries. Copper (nickel filter) and iron (manganese filter) radiation were used. For copper radiation, an average wavelength of 1.5418 Å was used for large interplanar spacings, and a $K\alpha_1$ of 1.54050 Å was used for resolved lines at small spacings. Corresponding iron values were 1.9373 and 1.93597Å. Relative intensities were measured on a Welch Densichron. Where possible, lattice parameters for the fluorostannates were calculated using reciprocal lattice techniques as described by Azaroff and Buerger.[11]

Density.—Density was determined pycnometrically in triplicate, using benzene dried over sodium as the immersion liquid. The stability of the compound was checked by X-ray powder patterns taken before and after measurement.

Magnetic Susceptibility.—The magnetic susceptibility of the solid fluorostannates was determined by the Gouy method. The magnetic moment was corrected for the diamagnetic susceptibility contribution of the remaining atoms.

Infrared Spectra.—Infrared spectra were determined with a Perkin-Elmer Model 337 spectrophotometer, using Halocarbon and Nujol mulls, with sodium chloride and potassium bromide plates. Far-infrared spectra were obtained with a Beckman IR-11 spectrophotometer; the Nujol mulls were supported on high-density polyethylene plates.

Results

Pertinent data for the complexes prepared are given in Table I. Replacement of oxygen by fluorine proceeded satisfactorily in all cases at approximately 500°. In addition to the strontium hexafluorostannate listed, there is a high-temperature form which could be prepared by fluorination of the stannate or of the low-temperature form at 640°, confirming the report of this form by Klemm and Hoppe.[12] No measurements were made on this high-temperature form.

The alkaline earth fluorostannates are not so sensitive to water vapor as are the remaining compounds, although all react with water. If they are kept in a nitrogen atmosphere for several hours after fluorination, there is no immediate subsequent weight change. Samples exposed to air for only a short time show infrared absorption in the –OH region and gradual decrease in fluoride content even in dry storage. For example, the fluoride content of a sample of $MgSnF_6$, held in a polyethylene container for 6 months, decreased from 44 to about 30%. At that time, X-ray powder patterns showed no change, and infrared spectra indicated absence of hydroxyl groups, observations comparable to those noted by Kolditz and Preiss[13] during polymerization of tin hydroxyfluorides. The octafluoro complexes are difficult to handle because of their extreme sensitivity to moisture.

The relatively simple powder patterns of the alkaline earth hexafluorostannates led to the deduction of their unit cells using systematic indexing procedures. It was also possible to index the patterns of the cobalt, nickel, and zinc complexes. The unit cell data, as determined from the powder patterns, are summarized in Table II.[14]

Discussion

Wells[4] pointed out that fluorides with the general formula ABF_6 crystallize with structures which are basically of the NaCl or CsCl type, although few crystallize with the ideal cubic structure. Rhombohedral and tetragonal distortions are known. Of the six possible variants, Babel[3] listed four as significant, of which the following are representative examples[15]

	NaCl type	Space group	CsCl type	Space group
Cubic	$NaSbF_6$	Fm3m, O_h^5	...	...
Rhombohedral	$LiSbF_6$, $KOsF_6$	$R\bar{3}$, C_{3i}^2	$TlSbF_6$	$R\bar{3}m$, D_{3d}^5
Tetragonal	...	...	$KNbF_6$	$P\bar{4}c2$, D_{2d}^6

Although the radius of the A cation does not appear to be the sole determinant of the structure, comparisons of

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(14) The powder data have been deposited as Document No. NAPS-00028 with the ASIS National Auxiliary Publication Project, % CCM Information Sciences, Inc., 22 W. 34 St., New York, N. Y. 10001. A copy may be secured by citing the document number and by remitting $1.00 for microfiche or $3.00 for photocopies. Advance payment is required. Make checks or money orders payable to: ASIS-NAPS.

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TABLE II

UNIT CELL AND DENSITY DATA

Compound	Type[a]	Z[b]	Unit cell constants, Å	Density, g/cm³ obsd	Density, g/cm³ calcd
$MgSnF_6$[c]	R	1	$a = 5.47$ ($\alpha = 58.1_2°$)	3.75	3.85
$CaSnF_6$	FC	4	$a = 8.30_4$	3.15	3.16
$SrSnF_6$	FC	4	$a = 8.62_2$	3.39	3.32
$BaSnF_6$[d]	R	1	$a = 4.93$ ($\alpha = 97.1_8°$)	4.99	5.25
$CoSnF_6$	H	3	$a = 4.94_3$, $c = 16.42$	4.21	4.18
$NiSnF_6$	H	3	$a = 4.91_7$, $c = 16.30$	4.09	4.25
$CuSnF_6$	?	...	...	4.69	...
$ZnSnF_6$	H	3	$a = 4.93_5$, $c = 16.42$	4.33	4.29
$CdSnF_6$	?	...	...	4.82	...
$MnSnF_8$	?	...	...	4.21	...
$PbSnF_8$	?	...	...	5.86	...

[a] R, rhombohedral; FC, face-centered cubic; H, hexagonal. [b] Number of formula weights per unit cell. [c] On hexagonal basis, $a = 5.31_0$ Å, $c = 13.57_8$ Å, $Z = 3$. [d] Given as hexagonal, $a = 7.40$ Å, $c = 7.40$ Å [R. Hoppe, *Rec. Trav. Chim.*, **75,** 569 (1956)] and as rhombohedral, $a = 4.97$ Å, $\alpha = 96.4°$ [B. Cox, *J. Chem. Soc.*, 876 (1956)].

TABLE III

INFRARED SPECTRA

Compound	ν_3[a]	Unassigned bands
$MgSnF_6$	622	376, 240, 169
$CaSnF_6$	609	228
$SrSnF_6$	591	259, 243, 163
$BaSnF_6$	573	285, 241, 201, 109
$CoSnF_6$	599	314, 240, 210, 184, 163
$NiSnF_6$	611	329, 245, 218, 171
$ZnSnF_6$	602	305, 239, 197, 158
$CuSnF_6$	605	340, 242, 213, 193, 176, 150
$CdSnF_6$	594	291, 227, 170, 134
$MnSnF_8$	?	585, 322, 251, 227, 179
$PbSnF_8$	?	503, 137

[a] Values in cm^{-1}; determined from center of band width at half-peak height.

the alkaline earth ionic radii suggest that $MgSnF_6$ would resemble $LiSbF_6$ ($R\bar{3}$), $CaSnF_6$ and $SrSnF_6$ would resemble $NaSbF_6$ (Fm3m), and $BaSnF_6$ would resemble $KOsF_6$ ($R\bar{3}$) or $TlSbF_6$ ($R\bar{3}m$). The powder patterns can be satisfactorily indexed on this basis, and the calculated and observed intensities of the reflections agree well for the lines measured. These intensities are associated primarily with the heavy-atom positions, and powder data are inadequate to determine precisely the locations of the fluorine atoms.

The similarity of cobalt, nickel, and zinc ionic radii to the magnesium ion radius suggests that the fluorostannates of these metals might also have the $LiSbF_6$ structure. Although hexagonal cells of similar size were found for these three complexes, satisfactory reduction to the appropriate rhombohedral cells was not possible. Copper and cadmium hexafluorostannates, with larger A cations, do not fit a recognizable pattern, nor do the manganese and lead complexes. Single-crystal studies of the complexes should be carried out, if such crystals can be obtained, since only in this way can the fluorides be located satisfactorily.

A large number of stretching frequencies for potassium fluorometalates of the type A_2BF_6 have been determined by Peacock and Sharp[16] in the region 1000–400 cm^{-1}. Two infrared-active vibrations, ν_3 and ν_4, would be expected for an octahedral molecule of symmetry O_h. In most cases ν_4 fell below the range studied, and the ν_3 vibrations were between 600 and 550 cm^{-1}, with that for SnF_6^{2-} at 552 cm^{-1}. As shown in Table III, the six-coordinate complexes exhibit a band in this region, indicating the presence of the SnF_6^{2-} anion in these compounds.

In the solid the SnF_6^{2-} ion may be in a site of symmetry lower than O_h, resulting in the splitting of a band representing a degenerate fundamental and in other modes becoming active. This has been discussed by Brown, *et al.*,[17] for some hexahalogenometalates(IV), using the approach outlined by Halford.[18] In general, spectra from the mulls were poorly resolved, with broad bands, making unequivocal interpretation difficult. The possibility of interaction with the mulling agent was considered, but the spectra show no absorption bands between 700 and 4000 cm^{-1}.

The $R\bar{3}$ (C_{3i}^2) space group of the $LiSbF_6$ structural type, indicated for $MgSnF_6$, assigns C_{3i} (S_6) symmetry to the SnF_6^{2-} ion site in the crystal. This requires six infrared-active fundamentals, in three frequency ranges. Of the three major bands observed, two are distinctly asymmetric. The middle band (376 cm^{-1}) lies higher than might be expected in comparison with the other alkaline earth fluorostannates, although in $LiSbF_6$ the ν_4 band is found at 350 cm^{-1}.[19]

Barium hexafluorostannate, with the $KOsF_6$ ($R\bar{3}$) or $TlSbF_6$ structure ($R\bar{3}m$ (D_{3d}^5), with D_{3d} site symmetry), should have six or five active fundamentals, respectively, in three ranges. Three bands are observed in the region 200–700 cm^{-1}, but it is not possible to distinguish between the two structures, which differ only by a scarcely perceptible displacement of fluorine atoms. The spectrum is essentially the same as that reported for $BaGeF_6$ ($R\bar{3}m$) by Griffiths and Irish,[20] without, however, a band corresponding to that at 452 cm^{-1}, also not detected by Brown, *et al.*, in K_2GeF_6, which has the same site symmetry.

The two cubic fluorostannates, $CaSnF_6$ and $SrSnF_6$, show distinct spectral differences. Two broad bands are observed for $CaSnF_6$, characteristic of the Fm3m (O_h^5) space group with the SnF_6^{2-} ion in O_h sites. The higher frequency band (ν_3) is not completely symmetric, and similar unexplained asymmetry has been noted in the ν_3 band of cubic hexafluorosilicates.[19] A small shoulder at 305 cm^{-1} occurs also in $SrSnF_6$, which otherwise shows marked similarity to the spectrum of trigonal K_2GeF_6, with D_{3d} site symmetry.[17,20] There is slight asymmetry of the ν_3 band (591 cm^{-1}), and the ν_4 band shows the same contours and splitting (259, 243 cm^{-1}), although at somewhat lower frequency. This indicates that the site symmetry is lower than O_h and that a more suitable space group might be Pm3m (O_h^4) in which the SnF_6^{2-} ions occupy D_{3d} site positions, al-

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though the C_{3i} site symmetry in the Pn3 (T_h^3) space group would probably be indistinguishable from this. These two groups have been proposed for $CaSn(OH)_6$.[21,22]

No pertinent corresponding conclusions can yet be drawn about the remaining complexes.

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The Solution of Sodium Alloys of Some Post-Transition Metals in Molten Sodium Halides. Evidence for Anions of Bismuth and Antimony[1]

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The solutions of sodium alloys of gold, thallium, tin, lead, antimony, and bismuth particularly in the molten solvent NaCl–NaI (37:63) have been examined as a function of alloy composition and temperature. The first four show relatively small solubilities of the heavy metal, ≤1.3 mol % at 700 or 800°. A larger, specific solution of $Na_{3.0}Bi$ occurs with metal phases containing 25–30 atomic % bismuth: 2 mol % at 600° and 27 mol % at 1000°. The solubility data are reasonably consistent with the alloy and melt solute species Na_3Bi plus the substantially less soluble NaBi. Freezing point studies for Na_3Bi in NaCl–NaI, NaI, and KI solvents suggest the simple ionic species Na^+ and Bi^{3-} are formed in dilute solution. The solubilities for the sodium–antimony system are substantially larger, about 11 mol % $Na_{2.5}Sb$ in equilibrium with Na_3Sb(s), 2 mol % Na_2Sb with NaSb(l), and 1.3 mol % $Na_{0.37}Sb$ with $NaBi_3$(l), all at 608°. Cryometric data for Na_3Sb and NaSb in sodium iodide suggest the presence of Sb^{3-} and Sb_3^{3-} anions in the melt, respectively. The dependence of solubility on solvent anion and on the heavy metal in the alloy or intermetallic compound are also considered.

Introduction

Although unusual lower oxidation states and bonding of cations are often found for solutions of the post-transition metals in their molten salts and related systems,[2–4] very little is known about *anions* of these elements even though some of these border on recognized, anion-forming nonmetals. In addition to the pioneering work of Zintl and coworkers[5] in liquid ammonia, there are the qualitative observations by Heymann and Weber[6] that sodium alloys with bismuth, antimony, tin, and gold (but not lead, cadmium, or thallium) dissolve to some extent in molten sodium bromide. Recently, the solution of Li_3Bi in an LiCl–LiF mixture (0.05 mol % at 600°, 2 mol % at 1000°) has also been reported[7] as well as spectral data for solutions of some alkali metal tellurides (M_2Te) in molten alkali metal halides.[8] The present paper reports more quantitative solubility data in the lower melting NaCl–NaI eutectic composition for many of the alloy systems studied by Heymann and Weber, including temperature and composition dependences for the more interesting antimony and bismuth systems. Cryometric data in several melts pertaining to nature of the solutes have also been obtained.

Experimental Section

Materials.—The bismuth (reactor grade), antimony (Bunker Hill), tin (Vulcan Materials), and thallium (American Smelting and Refining), all stated to be 99.999%, plus lead (American Smelting and Refining, 99.99+%) were purified by fusion in Vycor containers under dynamic vacuum (≤10^{-5} torr), a process which either volatilized or brought to the surface nonmetallic impurities. Gold (99.97%, J. Bishop) was used as received. The hydroxide was cut from sodium chunks (Baker, 0.0045% impurities) in the drybox and only the clean center pieces were used for alloy preparations. All compositions with lead, tin, and thallium are sufficiently low melting that they could be prepared by fusion of the weighed components in evacuated, sealed Pyrex or Vycor containers without attack, after which the liquid was poured to the other end of the tube to leave any oxide behind on the glass walls. The same technique was used to obtain a composition near Na_4Au which was then heated with additional gold in sealed tantalum tubes. Tantalum was used for all preparations of the higher melting antimony and bismuth compositions. The actual composition of all alloy samples used was determined by direct analysis either before or after equilibration with melt. In the former case the equilibrium alloy composition was calculated from the initial amounts of salt and known alloy and the solute content of the melt.

The NaI–NaCl eutectic (63 mol % NaI, mp 570°[9]) was made up from Mallinckrodt and Baker reagent grade salts, 0.05% impurities and 99.8% respectively, which were dried in Vycor containers under dynamic vacuum, first at room temperature and then gradually up to the fusion point. The salts used for the antimony solubility studies were also vacuum sublimed separately, though this precaution did not appear to affect the results appreciably.

Solution Studies.—The metallic systems which show appreciable solution in the molten eutectic readily attack Vycor or fused silica. Therefore, all solution measurements were carried out in sealed tantalum apparatus. As described earlier,[10] equilibrations

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