

THE BAND SPECTRUM OF GERMANIUM MONOIODIDE

WHEREAS a large number of band systems of the spectra of diatomic germanium fluoride, chloride and bromide molecules have been known, the spectrum of germanium monoiodide has not been known till recently. Oldershaw and Robinson¹ studied the absorption spectrum

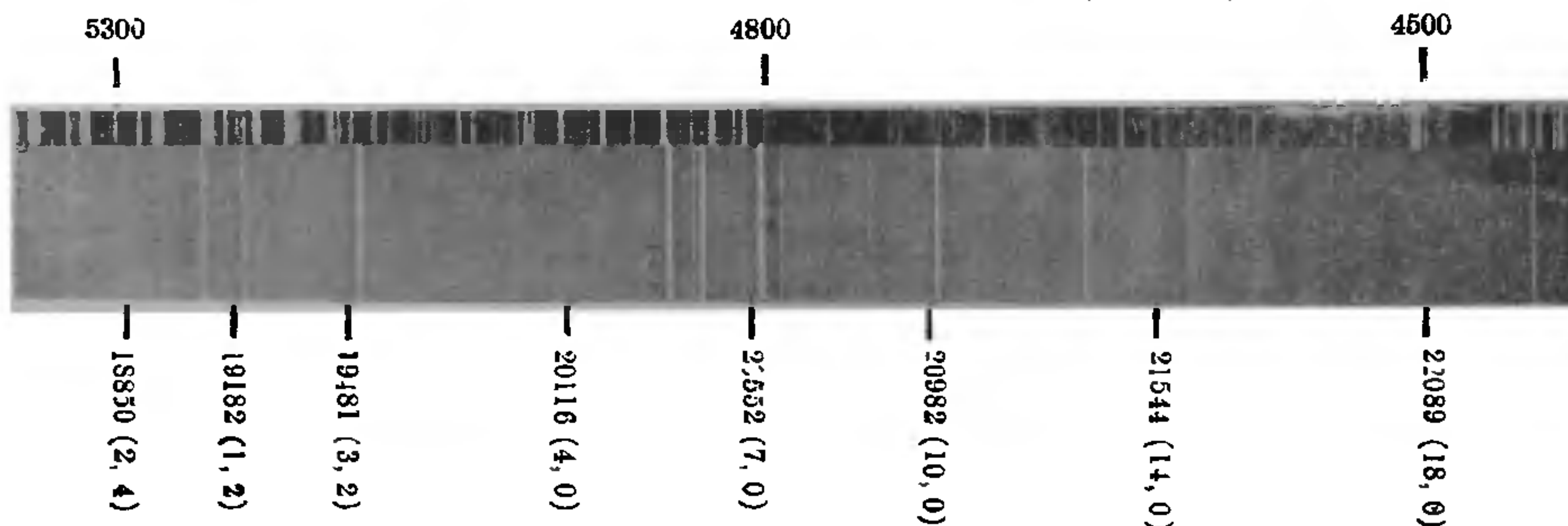


FIG. 1

of germanium monoiodide employing flash photolysis technique and reported a discrete band system in the ultraviolet region. In the course of our recent investigations on the halide spectra of IVA group elements a characteristic emission band spectrum attributable to germanium monoiodide molecule has been discovered in the visible region. The spectrum has been observed by exciting a mixture of germanium metal and iodine vapours by a R.F. discharge from a $\frac{1}{2}$ K.W. oscillator. The colour of the discharge is light yellow. Spectra recorded on Hilger 2-Prism Glass-Littrow and Fuess spectrographs revealed a series of red degraded bands extending from λ 6000– λ 4500. Figure 1 is the reproduction of the spectrum in the region λ 5300– λ 4500 recorded on the Fuess spectrograph. The wave-numbers of some of the prominent bands together with their vibrational assignments are shown. All these bands are analysed as belonging to a single system whose vibrational quantum formula is derived as follows:

$$\nu(v', v'') = 19566.9 + 151.2 \left(v' + \frac{1}{2} \right) - 0.44 \left(v' + \frac{1}{2} \right)^2 - 245.5 \left(v'' + \frac{1}{2} \right) + 0.50 \left(v'' + \frac{1}{2} \right)^2.$$

The near equality of the vibrational frequency $\omega e'' = 246.1 \text{ cm.}^{-1}$ for the ground state as determined from the absorption work by Oldershaw and Robinson with the value $\omega e'' = 245.5 \text{ cm.}^{-1}$ obtained in the present work supports the view that the emitter of this visible system is the

GeI molecule. For some of the bands with high v' values on the short wavelength side, the germanium isotopic effect has been detected. By analogy with similar band systems observed in the other homologues GeCl_2 , GeBr_2 , SiI_2 and SnI_2 molecules the transition involved in the emission of this new band system may be identified as $A^2\Sigma^+ - X^2\Pi_{1,2}$ of the GeI molecule.

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1. Oldershaw, G. A. and Robinson, K., *Trans. Faraday Soc.*, 1968, **64**, 2256.
2. Babu Rao, K. and Haranath, P. B. V., *J. Phys. B.*, 1969, **2**, 1080.
3. — and —, *J. Phys. B.*, 1962, **2**, 1335.
4. Lakshminarayana, A. and Haranath, P. B. V., *Ibid.*, 1969, **38**, 136.
5. Adinarayana Murty, A. and Haranath, P. B. V., *Ibid.*, 1969, **38**, 211.

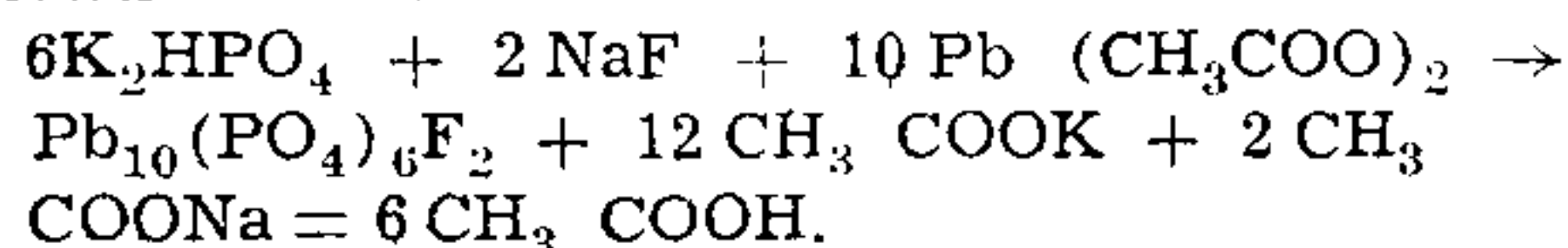
LEAD FLUORAPATITE, $\text{Pb}_{10}(\text{PO}_4)_6\text{F}_2$

THE lead apatites are a group of inorganic compounds which can be represented by the general formula $\text{Pb}_{10}(\text{PO}_4)_6\text{Z}_2$ where $\text{Z} = \text{Br}^-$, Cl^- , F^- or OH^- . They have the hexagonal structure with space group $\text{P6}_3/m(\text{C}_6^2h)$.

A new method of preparing lead fluorapatite, $\text{Pb}_{10}(\text{PO}_4)_6\text{F}_2$, which has proved quite effective is reported here. The formation of this compound was described by Merker and Wondratschek¹ who prepared it by sintering stoichiometric amounts of lead phosphate, $\text{Pb}_3(\text{PO}_4)_2$, and lead fluoride, PbF_2 , at 660°C . in platinum tubes. The unit cell dimensions were given as: $a = 9.76 \text{ \AA}$, $c = 7.29 \text{ \AA}$, and $c/a = 0.746$.

The lead fluorapatite of this investigation was prepared by using stoichiometric quantities of K_2HPO_4 , NaF , and $\text{Pb}(\text{CH}_3\text{COO})_2$. All chemicals were of laboratory grade and were

used as supplied. A solution of K_2HPO_4 (1.80 gm./100 ml.) was mixed with a solution of NaF (0.14 gm./50 ml.). This mixed solution was added dropwise to a continuously stirred solution of $Pb(CH_3COO)_2$ (5.40 gm./100 ml.). The temperature of the reacting mixture was held at 100° C. The stirring was continued for about 2½ hours and the lead fluorapatite which had formed was removed by filtration, washed with distilled water, and dried at 100° C. The proposed reaction is shown below :



The compound starts decomposing above 400° C., but it does not melt completely upto 1021° C. It is insoluble in water and several known solvents but very slightly dissolves in nitric acid. Analysis : calculated for $Pb_{10}(PO_4)_6F_2$; Pb = 77.30%, $PO_4 = 21.27\%$. Found : Pb = 77.25%, $PO_4 = 21.10\%$.

Infrared spectrum was run from 400 to 1200 cm^{-1} on Perkin-Elmer 337 Infrared spectrophotometer using KBr pellets. Major peaks in the spectrum were : 530, very strong, 560 very strong ($\nu_4 PO_4^{3-}$), 935 to 1,065 very strong, broad ($\nu_3 PO_4^{3-}$).

The X-ray powder diffraction pattern was taken on a CGR (France) instrument in a Seeman-Bohlin camera using $CoK\alpha$ radiation ($\lambda 1.77892 \text{ \AA}$). The powdered apatite and small amount of calcite used as an internal standard were thoroughly ground in an agate mortar and spread over a mount of polystyrene foil. The following lattice constants resulted from the cell based on the space group C_{6h}^{2h} : $a = 9.77 \text{ \AA}$, $c = 7.30 \text{ \AA}$ and $c/a = 0.747$. The results of measurements clearly show that the lead fluorapatite prepared by this process is identical with that synthesized by the method of Merker and Wondratschek.¹

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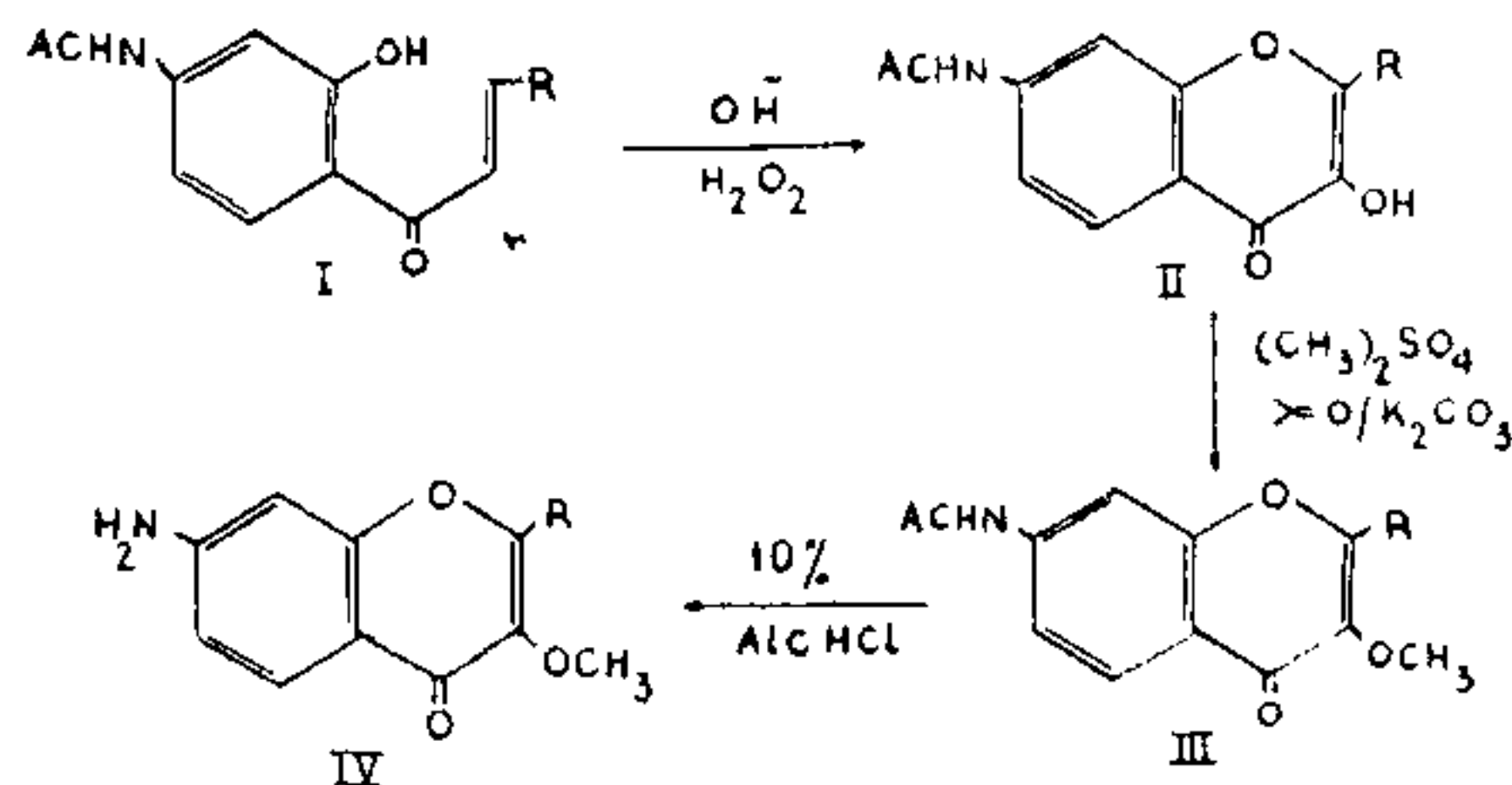
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1, Merker, L. and Wondratschek, H., *Z. anorg. allgem. Chem.*, 1959, 41, 300.

SYNTHESIS OF 3-METHOXY-7-AMINO FLAVONES AS POTENTIAL BACTERICIDES

GEIGER AND CON¹ AND KAMODA AND ITO² have shown that $\alpha : \beta$ -unsaturated ketones exhibit bacteriostatic activity. Bacteriostatic action of $\alpha : \beta$ -unsaturated ketones has been found to increase with the introduction of halogen and hydroxy substituents.^{3,5} Since 4-amino salicylic acid is well known for its anti-tubercular properties, 7-amino flavone was prepared and found to inhibit the growth of *Mycobacterium tuberculosis*.⁶ Subsequently, a number of 7-amino flavones were prepared in our laboratories and tested for their bacteriostatic activity.⁷ The synthesis of amino and halogeno substituted flavonols has, therefore, been taken up with a view to test their bacteriostatic activity.

Condensation of *p*-acetamino-*o*-hydroxy acetophenone with benzaldehyde, 2-methoxy, 2 : 4-dimethoxy, 3 : 4-dimethoxy, 2 : 4-dichloro, and *p*-chloro benzaldehydes in 30% aqueous alcoholic KOH led to the formation of the corresponding chalcones (I) in 80% yield. The chalcones on subjecting to Algar-Flynn-Oxidation (A.F.O.)⁸ gave the corresponding 7-acetamino-3-hydroxy flavones (II) in better yields at room temperature rather than at 0° C. The flavonols thus obtained have been methylated (III) and hydrolysed using 10% alcoholic hydrochloric acid to obtain the corresponding 7-amino-3-methoxy flavones (IV). The compounds synthesised together with their melting points are listed in Table I.



All the compounds listed in Table I have been tested for their activity against *Bacillus coli*, and *Staphylococcus aureus* using the tube dilution method.⁹ None of them showed activity against *S. aureus* but exhibited activity against *E. coli* at a concentration of 10,000 dilution. Of these, 2'-hydroxy-4'-acetamino chalcone and 3-hydroxy-3'-4'-dimethoxy-7-acetamino flavone were active at 1,00,000 dilution.