

LETTERS TO THE EDITOR

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APPLICATION OF X-RAY METHODS TO THE CONFIRMATION OF THE IDENTITY OF ORGANIC COMPOUNDS

THE identity of organic compounds is usually shown by the undepressed melting point of mixtures of substances and of mixtures of their similar derivatives. We have used X-ray methods of crystal structure analysis for the confirmation of the identity of 2:4 dibromo-6-nitro resorcinol-3-methylether obtained by one of us (M. S.) by bromination and nitration respectively of 5-nitro-2-hydroxy-4-methoxy-benzaldehyde and 3:5 dibromo-2-hydroxy-4-methoxy-benzaldehyde. In both cases, on recrystallisation, fibrous acicular crystals were obtained. They were both found to be orthorhombic with one of the crystallographic axes coincident with the length of the needles. Rotation photographs of crystals obtained by both methods were taken, the length of the acicular crystals being parallel to the axis of rotation, using copper K_{α} radiation and a cylindrical camera of radius 32.03 mm. The identity period obtained from these photographs are $4.13 \pm 0.02 \text{ \AA}$ for the product obtained by nitration

and $4.11 \pm 0.02 \text{ \AA}$ for the other. The photographs were also identical in all respects, all the corresponding spots coinciding exactly on superposition of the photographs. Accurate measurements of the co-ordinates of the more prominent spots made on one photograph were found to be in agreement with those on the other within the limits of errors of measurement. These results confirm the identity of the two products obtained as indicated above.

S. RAMA SWAMY.

M. SESHAIYENGAR.

Department of Physics, Central College,
and

Intermediate College,

Bangalore,

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A NEW BAND-SYSTEM OF BORON MONOXIDE

A PRELIMINARY note on some continuous diffuse bands (fluctuation bands) obtained in the spark between glass electrodes reported the presence of discrete bands among some of the fluctuation bands.¹ An analysis of these discrete

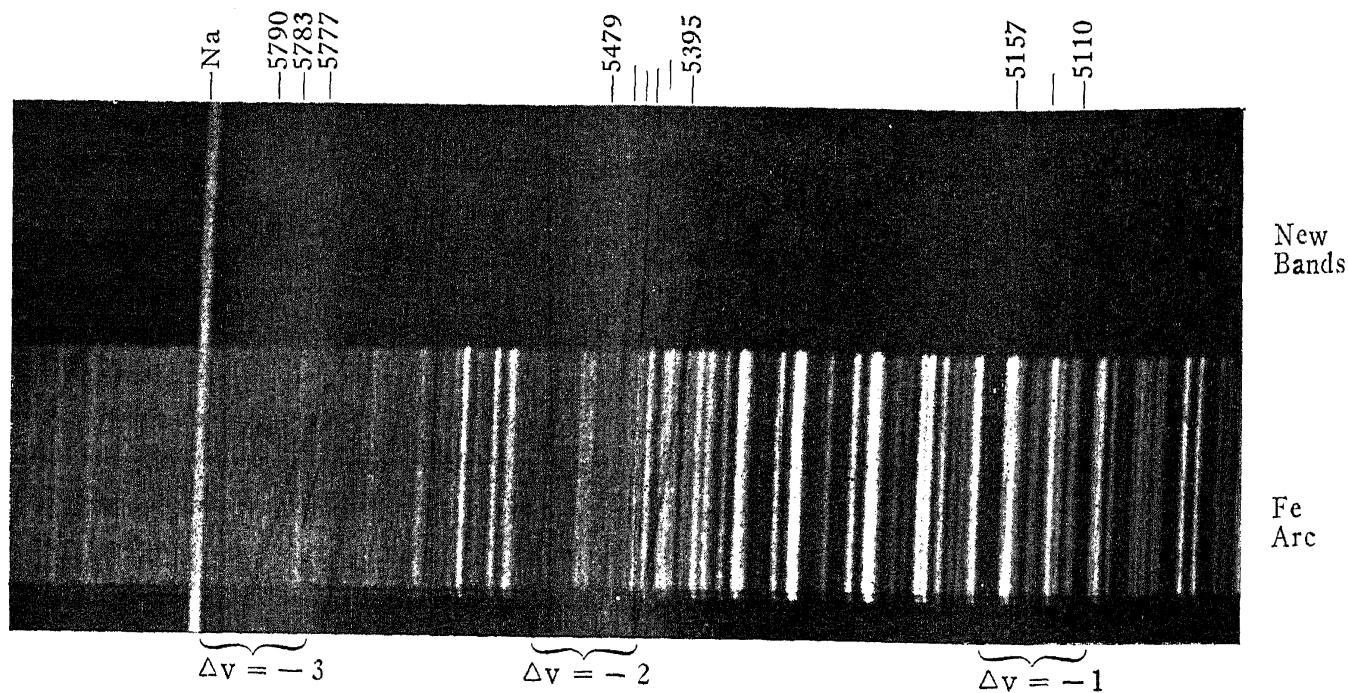


FIG. 1

bands has now been done, which shows that they form a new electronic system in the BO molecule. As a result of this analysis it has been further found out that the diffuse fluctuation bands are not due to SiO_2 , but are the well-known so-called boric oxide bands.²

It will be seen from Fig. 1, which is an enlarged reproduction of a spectrogram of the radiation from a Bunsen burner fed with boric acid, taken on a constant deviation glass spectrograph, that there are at least three groups of bands which form three sequences. These when arranged in the $v'v''$ array yield the following $\Delta v''$ values given in Table I, where the $\Delta v'$ values³ for the β -bands of B^{11}O are also included.

TABLE I

Vibrational Term Differences

Levels	0-1	1-2	2-3	3-4	4-5	5-6	6-7
Initial of β bands of $\text{B}^{11}\text{O} = \Delta v'$	1259	1237.8	1217.4	1198.6	1179.1	1160.7	—
Final of the new system = $\Delta v''$	—	—	1225	1206	1174	1162	1142

The agreement between the two sets of values indicates first, that the electronic levels of which these are the vibrational term differences are identically the same and secondly, that the first term difference observed in the new bands is already the one between the second and the third vibrational levels of BO in this common electronic state. On this basis the following expression is derived for the wave-numbers of the band heads of the new system:—

$$\nu_{\text{head}} = 21005.6 + (1200 v' - 15.4 v'^2) - (1266 v'' - 10 v''^2).$$

The O,O band has not been observed but the v' numbering also seems certain. The expression further shows that the final vibrational function $(1266 v - 10 v^2)$ of the new system is, within the limits of allowable error, the same as the initial of the β bands of B^{11}O $(1268.8 v - 9.98 v^2)$. The new electronic level is thus located at $\nu = 63870.8 \text{ cm.}^{-1}$ (21005.6 plus 42865.2 the origin of β bands) or 7.9 e.v.

These bands are not usually obtained in the method of production (active nitrogen plus BCl_3 vapour) employed for the development of the α , β and combination band-systems of BO. Also the latter band-systems are not developed, except for possible traces of some of them, either in the boric acid flame or the spark discharge. The new bands, however, always

accompany the diffuse fluctuation bands, the so-called boric oxide bands, which also appear to be due to BO molecule. These and other considerations pertaining to the diffuse bands indicate that the initial state involved in the new band-system is a Σ , possibly a quartet Σ level, arising out of B ($2s^2 2p^2, ^4P$) and O ($2s^2 2p^4, ^1D$). There is further reason to think that probably such a state of BO is directly reached when the molecule is obtained from B_2O_3 .

It is remarkable, however, that the spectrograms which show these diffuse fluctuation bands in the glass spark do not show any trace of the resonance lines of boron. It is evident that for detection of boron in the form of B_2O_3 or boric acid, these bands are much more sensitive than the *raies ultimes* of boron. The glass used was supposed not to contain any chemically traceable boron.

Details regarding these and other points will be published elsewhere. I am grateful to Prof. R. K. Asundi for helpful discussion and continued interest in the work.

NAND LAL SINGH.

B/1 Quarters,
Benares Hindu University,
June 12, 1942.

¹ *Proc. Ind. Sci. Congress, Benares, 1941, 29, Part III.*

² *Handbuch der Spectroscopic, 1910, Band V, 138.* (Kayser).

³ *Phys. Rev., 1925, 25, 59.*

A NOTE ON THE ISOLATION OF THREE NEW BITTER PRINCIPLES FROM THE NIM OIL

THE active constituents of the Nim oil have interested a number of workers since Chatterji and Sen¹ reported the isolation of the so-called 'margosic acid' from it, in 1919. The investigations on this problem have, however, led to findings of a very conflicting character. Thus the margosic acid, which was considered to be the active principle of Nim oil was later shown to be mainly a mixture of fatty acids.² In 1923, Watson and co-workers,³ isolated from the soap lye of the oil a sulphur-free crystalline bitter acid

'margoso-pierin', in a yield which works out to 0.012 to 0.017 per cent. on the weight of the oil and amorphous bitter acids in a yield of ca. 0.15 to 0.24 per cent. Sen and Banerji noted the isolation of a sulphur containing acidic bitter principle from the aqueous extracts of the oil (yield not mentioned). More recently Quadrat-i-khuda and co-workers⁴ communicated the isolation of the sulphur-containing essential oil and an amorphous, water-soluble bitter principle from the aqueous extracts of the oil, after previously subjecting it for long periods to steam distillation for removal of the steam volatile products.

The methods employed by Watson and co-workers as well as by Q. Khuda and co-workers for the isolation of the active principle, appear to be of too drastic a character to ensure the isolation of the bitter principle in its native condition. In view of the growing importance of the Nim oil as a commercial product and the long established uses of Nim in the indigenous systems of medicine as a bitter tonic, an anti-malarial and anthelmintic, and as a cure for syphilitic conditions and a variety of skin diseases, a re-investigation of the active principle of the oil was considered of interest, as part of a general scheme of research for establishing its industrial uses.

Working on a so far unexplored plan of exclusively mechanical separation of the various constituents of the Nim oil, the following well-defined bitter constituents have been isolated from it, the total bitter constituents being obtained in an industrially workable yield of ca. 1.2 per cent. as against 0.24 per cent. reported by Watson and co-workers:

- (1) A sulphur-free, neutral, water-insoluble, colourless crystalline product melting at 205° C. and provisionally named as 'NIMBIN'. Yield—0.1 per cent. on the weight of the oil.
- (2) A bitter principle of similar characteristics as 'NIMBIN' melting at 192° C., which has been provisionally named as 'NIMBININ'. Yield—0.01 per cent. on the weight of the oil.