the exception of illinium and thulium and concluded that samarium was the only one of the group whose radioactivity persisted after purification.[8] As thulium is classified in the first group, it also may possess radioactive properties of a magnitude comparable with that of samarium. Hence, a study of the radioactivity of thulium preparations is well worth investigating.

CONTRIBUTION FROM THE
DEPARTMENT OF CHEMISTRY
COLUMBIA UNIVERSITY,
NEW YORK CITY

RECEIVED JULY 16, 1935

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[8] G. von Hevesy and M. Pahl, *Nature*, **131**, 434 (1933); *ibid.*, *Z. physik. Chem.*, **A169**, 147 (1934).

COMMUNICATIONS TO THE EDITOR

ATTEMPTED PREPARATION OF VANILLOYLFORMIC ACID

Sir:

In an attempt to repeat the oxidation of apocyanin to vanilloylformic acid with nitrobenzene in alkaline solution as described by H. O. Mottern [THIS JOURNAL, **56**, 2107 (1934)] no action whatever was observable under the conditions described by the author. Nitrobenzene was recovered quantitatively from the reaction mixture and neither aniline nor azobenzene was detectable by qualitative means.

Difficulty was also experienced in repeating the author's description of the preparation of apocyanin by means of the Fries reaction. The aluminum chloride double compound with guaiacol acetate is quite solid at the temperatures described (0–5°). Before a third of the required amount of aluminum chloride has been added, the mass is so hard as to be impenetrable to a stirring rod, making it impossible to incorporate the balance. The apocyanin used was prepared by the method for low temperature Fries isomerizations described by Baltzly and Bass [*ibid.*, **55**, 4293 (1933)] which differs from the authors in the use of nitrobenzene as a solvent.

FARMINGDALE, L. I. W. E. BARCH

RECEIVED MARCH 12, 1935

NOTE BY THE EDITOR

After the receipt of this Communication a repetition of the oxidation of apocyanin to vanilloylformic acid with nitrobenzene in alkaline solution as described by H. O. Mottern was attempted by a member of the Editorial Board and independently by the Director of Research of a firm manufacturing vanillin on a large scale. Both were unsuccessful; their concordant conclusion is expressed by the latter's statement: "The synthesis of vanillin cannot be carried out following the disclosures of the paper published in THIS JOURNAL, 1934, page 2107."

OCTOBER 10, 1935 ARTHUR B. LAMB, *Editor*

THERMAL DECOMPOSITION OF $CrO_4 \cdot 3NH_3$

Sir:

Some unusual observations recently made in this Laboratory, and which we hope to study in greater detail, should prove of considerable general interest. While attempting the preparation of the tetroxide of chromium, CrO_4, by deammonating $CrO_4 \cdot 3NH_3$ (I) [made according to Riesenfeld, *Ber.*, **38**, 4070 (1905)], the following was noted. (1) Compound (I) loses practically no ammonia upon standing for four months over concentrated sulfuric acid *in vacuo* and at room temperature. (2) If the pressure in a tube containing some (I) is reduced to less than 1 mm. and the tube, still attached to the vacuum line, is immersed in a bath whose temperature may be controlled, a vigorous reaction ensues at 120 ± 10°. Particles of (I) become incandescent and dart about in spectacular fashion. The action is reminiscent of the thermal decomposition of ammonium dichromate and also of certain models that have been constructed to portray molecular motion in gases. (3) The solid residue of the above mentioned decomposition of (I) is composed of microscopic, elongated square prisms of black $CrO_3 \cdot NH_3$ (II). Analysis of (II) for ammonia was made by distilling into standard acid and for chromium by forming lead chromate. The re-