

For methane, three values were computed, depending on which line spacing one assumes to be representative of the moment of inertia. They are 44.1, 42.3 and 42.0; for  $I = 10.2, 5.66$  and  $5.17 \times 10^{-40}$ . The last corresponds to the spacing most accurately measured (Raman effect)<sup>5</sup> and, for various reasons, is probably the correct one. In fact, the agreement with the observed entropy<sup>6</sup> 42.5 may be adduced as additional evidence for the reliability of this choice.

These results indicate that the actual calculation of thermodynamic equilibria<sup>7</sup> from band spectra data is not to be much longer limited to reactions between molecules having a maximum of two atoms.

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#### 2,4,5-TRIMETHOXYBENZOIC ACID: A DERIVATIVE OF DEHYDRODEGUELIN *Sir:*

It has been shown by LaForge and Smith<sup>1</sup> that, through a series of reactions, the rotenone molecule may be ruptured in a manner which yields derric acid,  $C_{12}H_{14}O_7$ . Derric acid represents the half of the rotenone molecule which contains the methoxyl groups, but as yet the exact nature of the acid is unknown.

Recently three other insecticides of the same type as rotenone, namely, deguelin, tephrosin and toxicarol, have been described.<sup>2</sup> It has also been shown that structural relationships exist common to the entire molecule of rotenone, deguelin and tephrosin;<sup>3</sup> but the fact which is of immediate interest is that the derric acid portion of the molecule is common to all three substances.

It is desired at this time to report in a preliminary way the results of some experiments which clarify to a considerable degree the problem of the derric acid portion of rotenone and allied materials.

Permanganate oxidation of dehydrodeguelin in acetone solution yields two hydroxy acids, one of which contains the two methoxyl groups present in the starting material and thus represents the derric acid portion of the molecule. This acid crystallizes in plates which may be roughly described as rectangular with notched ends and with constrictions in the center of their long axis. It melts at  $210^\circ$  with decomposition and gives an intense blue color with ferric chloride.

<sup>5</sup> Dickinson, Dillon and Rasetti, *Phys. Rev.*, **34**, 582 (1929).

<sup>6</sup> Randall and Mohammad, *Ind. Eng. Chem.*, **21**, 1048 (1929).

<sup>7</sup> Cf. Ludloff, *Z. Physik*, **57**, 227 (1929), for a general discussion of this problem.

<sup>1</sup> LaForge and Smith, *THIS JOURNAL*, **52**, 1091 (1930).

<sup>2</sup> Clark, *ibid.*, **52**, 2461 (1930); **53**, 313 (1931); **53**, 729 (1931).

<sup>3</sup> Clark, *Science*, **73**, 17 (1931); also a paper now being submitted to *THIS JOURNAL* for publication.

Analysis proved the material to be an hydroxydimethoxybenzoic acid. Methylation of the free hydroxyl group gave a trimethoxybenzoic acid which was shown by its melting point and derivatives obtained by bromination and nitration to be asaronic acid, 2,4,5-trimethoxybenzoic acid.

The hydroxydimethoxybenzoic acids which could give this acid are 2-hydroxy-4,5-dimethoxy-, 5-hydroxy-2,4-dimethoxy- or 4-hydroxy-2,5-dimethoxybenzoic acid. It is thought that because of the ferric chloride reaction given by the acid from dehydrodeguelin it is 2-hydroxy-4,5-dimethoxybenzoic acid. Work upon its synthesis is in progress, and it is hoped that a report upon its structure can be made shortly.

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#### SYNTHESIS OF GAS-METAL COMPOUNDS BY SPUTTERING

*Sir:*

In connection with a somewhat extensive study of cathodic sputtering [see *Phys. Rev.*, **32**, 649 (1928); **34**, 972 (1929); *Nature*, **126**, 204 (1930)], the writer has recently found that this process has remarkable possibilities as a method of chemical synthesis. This applies particularly to the formation of metal-gas compounds such as nickel and similar metals with hydrogen, nitrogen, etc. Nickel sputtered in nitrogen under rather special conditions of current, voltage and gas pressure gives a dark, somewhat metallic-looking film which shows on x-ray examination a crystal structure distinctly different from that of the pure metal. Heating to about 350° decomposes this compound, which gives off its gas and reduces to metallic nickel. Upon heating such a nitride film to 150° in hydrogen, ammonia is formed and in an amount which agrees reasonably well with the quantity of nitrogen absorbed in sputtering. Compounds of cobalt and iron with nitrogen have likewise been produced, as well as of nickel and hydrogen—this latter showing on crystal structure examination a lattice spacing some 6% larger than for pure nickel.

It seems certain then that we are dealing here with such unusual compounds as nitrides and hydrides of these metals. Moreover, they are formed, not at the expense of a difficult and special technique of synthesis [see A. C. Vournasos, *Compt. rend.*, **168**, 889 (1919)] but by a simple process of wide applicability, for the conditions encountered in sputtering, with the metal in the vapor state and the gas largely excited by the discharge, would seem to be ideal for the formation of a compound if this is chemically possible. Indeed one series of experiments yielded considerable evidence for the formation of compounds with even helium and