to the participation of the structure $\rangle\bar{C}—\dot{O}$ in the alkoxide.

The results on the isopropoxide confirm the prediction[1] that the hyperfine splitting constant of the ketyl radical $(CH_3)_2\dot{C}\bar{O}$ should be a few œrsteds smaller than that of the isopropanol radical $(CH_3)_2\dot{C}OH$.

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Determination of the Initial Formation Temperature of Nickel Ferrite Spinel

Little information is to be found in the literature[1,2] about the merits of different techniques which may be used for the detection of initial spinel formation temperatures. This communication compares a number of methods which have been used for determining the spinel formation of nickel ferrite, $NiFe_2O_4$.

A mixture of iron and nickel oxides, both of > 99·7 per cent purity, in the proportions appropriate to the stoichiometric formation of nickel ferrite, was prepared by the usual powder metallurgy techniques[3]. Magnetic susceptibility measurements, electrical resistivity measurements and X-ray diffraction measurements were carried out on samples of the mixture that had been calcined in air for 4 h at temperatures ranging from 400° to 1,260° C. The reaction was also followed by thermal gravimetric analysis and differential thermal analysis. From the results of these measurements shown in Fig. 1 and Table 1, it is apparent that the five techniques show a spread of about 100° C in the detected initial formation temperatures.

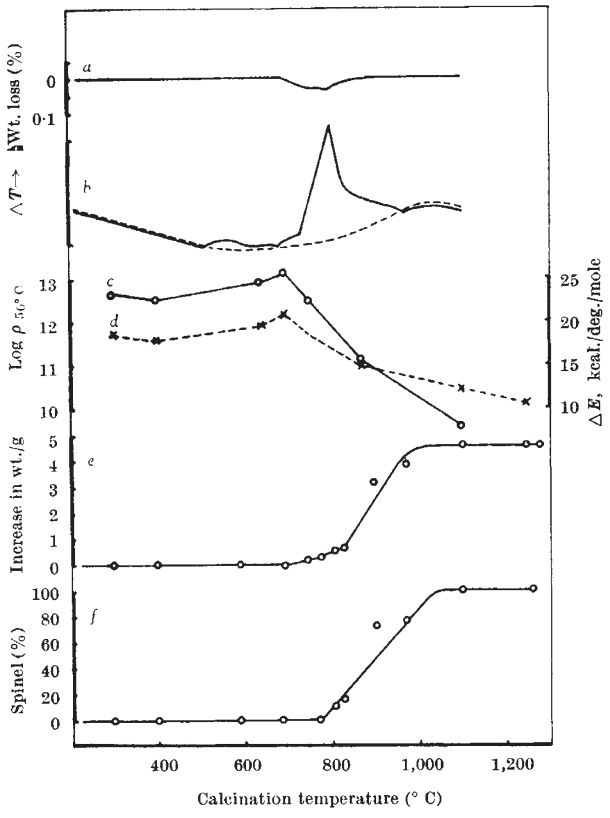

Fig. 1. Initial spinel formation temperature of nickel ferrite determined by: *a*, Thermal gravimetric analysis; *b*, differential thermal analysis (dotted line, estimated base line); *c*, electrical resistivity; *d*, activation energy (calculated from log $\rho_{50-100^\circ C}$); *e*, magnetic susceptibility (Gouy method); *f*, X-ray diffractometry. Samples for *c*, *d*, *e*, *f* were calcined for 4 h in an air atmosphere

Table 1. $NiFe_2O_4$ Spinel Formation Temperatures

Method	Initial spinel formation temperature (°C)	Complete spinel formation temperature (°C)
Thermal gravimetric analysis	690	900
Differential thermal analysis	730	965
Electrical resistivity	690	—
Activation energy	690	—
Magnetic susceptibility	750	1,100
X-ray diffractometry	780	1,100

The measurement of electrical resistivity appears to be the most sensitive method; the change from the defect conductivity of NiO and Fe_2O_3 to the electron conductivity of the spinel shows clearly at 690° C. In addition to the absolute value of resistance, the activation energy of conductivity has also been calculated and plotted, indicating a similar sharp peak.

The magnetic susceptibility and X-ray diffraction techniques, while not quite so sensitive as the electrical resistivity method, enable quantitative information to be obtained about the temperature of complete ferrite formation. All three methods suffer from the disadvantage of requiring lengthy sample preparation.

Both the thermal analytical techniques—thermal gravimetric analysis and differential thermal analysis—provide a much quicker method of measurement, but the results are only of a qualitative nature. In the analysis of the formation of nickel ferrite, differential thermal analysis is much more satisfactory than thermal gravimetric analysis, but for other ferrites, where a reduction occurs in the oxide mixture (for example, MnO_2 plus Fe_2O_3), thermal gravimetric analysis can provide both qualitative and quantitative data about the spinel formation process.

This work is being continued with other transition metal spinels of the formula MFe_2O_4, where M = Cu, Co, Mg, Mn and Zn, and the complete investigation will be published shortly.

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Effect of Substituents on Electrical Conductivity of Anils

This communication reports an attempt to approach the problem of electrical conductivity in organic solids from the point of view of classical organic chemistry. A series of anils derived from salicylaldehyde and various meta- and para-substituted anilines (Fig. 1) has been prepared and the electrical conductivities of compressed silver-coated polycrystalline pellets of each species have been measured. Most of these materials showed an observable dark conductivity under vacuum even at temperatures at which they were shown to be essentially non-volatile. It seems unlikely, therefore, that the conduction process could be described at these temperatures by a surface mechanism such as has been proposed by Eley *et al.*[1] for volatile organic crystals.