

DETECTION OF HO_{2} RADICALS BY THE E.P.R. METHOD

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Abstract

Full Text

PHYSICAL CHEMISTRY

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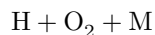
DETECTION OF HO₂ RADICALS BY THE E.P.R. METHOD

(Presented by Academician V. N. Kondrat'ev, May 20, 1961)

For a comparatively long time the existence of the HO₂ radical was postulated in many oxidation reactions. With the development of new experimental techniques it was shown that the HO₂ radical does indeed exist. Thus, in the work of Foner and Hudson (¹), the formation of HO₂ upon interaction of H atoms with molecular oxygen was demonstrated by means of a mass spectrometer. The H atoms were obtained in a discharge in H₂.

Fig. 1. E.p.r. spectrum of the HO₂ radical, frozen at 77° K

The study of elementary reactions of the HO₂ radical raises the problem of finding methods simpler than the mass-spectrometric one for the analysis of this radical. Such a simpler method proved to be the method of electron paramagnetic resonance (e.p.r.). In the literature there are no data in which an e.p.r. signal would unquestionably have been ascribed to the HO₂ radical. In our experiments the HO₂ radical was obtained by the interaction of H atoms with an O₂ molecule at room temperature under jet conditions at a total pressure of 60 mm Hg. The linear velocity of the jet was 90 cm/sec. The H atoms were obtained in a silent discharge in H₂ in an ozonizer-type tube. This tube was connected with the reaction vessel through a nozzle. Molecular oxygen was fed into the reaction vessel behind the nozzle. The dependence of the rate of formation of HO₂ on pressure indicates that the reaction is termolecular, i.e., proceeds in the volume. A detailed description of the apparatus and the results of kinetic studies of the reaction



will be published later. The HO₂ radicals were frozen out in a thin tube cooled with liquid nitrogen. Cooling with liquid nitrogen was carried out at a distance of 4 cm from the place where H was mixed with O₂. After the reaction had been carried out, the tube together with the cooling vessel was sealed off and

transferred to the instrument for recording the e.p.r. spectrum.* The e.p.r. spectrum was recorded on a 3-centimeter radiospectrometer with double modulation of the magnetic field, type $\text{M}-2$ (²). Figure 1 shows the appearance of the e.p.r. spectrum of the HO_2 radical obtained in the gas phase and frozen at 77°K . As is seen from Fig. 1, the signal has an asymmetric shape. The total width of the signal between points *A* and *B* is 23–26 oersted. The *g*-factor of the HO_2 radical practically coincides with the *g*-factor of diphenylpicrylhydrazyl.

* The e.p.r. spectra were recorded by G. A. Kapralova, to whom the authors express their gratitude.

Heating the vessel followed by cooling again to 77°K leads to the disappearance of the signal. In experiments without the addition of oxygen (blank experiments), no signal is observed. In parallel with recording the EPR spectrum, a chemical analysis of the reaction products was carried out. In experiments in which the EPR signal of the HO_2 radical was detected, after the tube was opened H_2O_2 was found. In blank experiments, in which no signal was observed, H_2O_2 was absent.

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- ² A. G. Semenov, N. N. Bubnov, *Pribory i tekhnika eksperimenta*, **1**, 92 (1959).

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