

From these preliminary results some features concerning the oxidation of perfluoropropylene seem of particular interest. It is tempting to correlate the 1:1 ratio of COF_2 and CF_3COF with the depolymerization mechanism as proposed for the oxidation of tetrafluoroethylene⁸. The infra-red spectrum of the fraction boiling at about -30°C suggests the presence of an epoxidic product together with perfluoropropionyl fluoride. However, the analysis of the liquid indicates a high O/C ratio which disagrees with the expected composition of the polyperoxide.

The large uptake of oxygen may be due to some secondary oxidation process, as, for example, for the attack of oxygen to CF_3 groups. Finally, it may be remarked that contrary to polymerization the oxidation of perfluoropropylene proceeds at a rate comparable with that of tetrafluoroethylene⁸.

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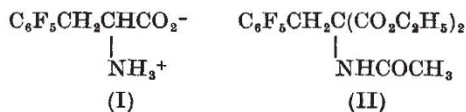
⁴ British patent 904,877 (1962) to du Pont de Nemours; U.S. appl., July 17 (1959) and June 10 (1960).

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Synthesis of Pentafluorophenylalanine

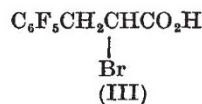
OUR interest in fluorine-containing α -amino-acids¹⁻³ has led us to an investigation of the chemistry of β -pentafluorophenylalanine (I). We wish to report here the synthesis of this novel amino-acid by two different routes. (In a personal communication, Dr. P. L. Coe, University of Birmingham, England, has informed us that he has prepared compound I in impure form by means of the azlactone route. Preliminary tests with several micro-organisms indicated that the amino-acid was inactive.)



In the method of choice, pentafluorobenzyl bromide⁴ reacts with sodiodiethylacetamidomalonate in dimethylformamide at 40° for 20 h to give a 44 per cent yield of the condensation product (II), m.p. 124° – 126° . Compound II (3.0 g) was heated under reflux for 8 h with 20 per cent hydrochloric acid to furnish the hydrochloride of I. This salt readily loses hydrogen chloride on crystallization from 95 per cent ethanol to give 1.4 g (73 per cent) of the free amino-acid I, m.p. 251° – 254° (uncorrected). Analysis: Calc. for $\text{C}_9\text{H}_5\text{F}_5\text{NO}_2$: C, 42.36; H, 2.37; N, 5.49 per cent. Found: C, 41.98; H, 2.63; N, 5.77 per cent. The amino-acid gives a strong positive ninhydrin test and its infra-red spectrum is consistent with the structure proposed. The weakly basic character of the amino group in I relative to the amino group in β -phenylalanine is reflected by the rapid loss of hydrogen chloride and by preliminary pK_a data. We attribute this effect in some measure to an interaction between an ortho fluorine atom and the NH_3^+ group across intramolecular space.

The second method involves the conversion of α -bromo- β -pentafluorophenylpropionic acid (III) (obtained from pentafluoroaniline and acrylic acid by means of the Meerwein arylation method⁵) to the α -azido compound⁶ and subsequent hydrogenolysis, using 10 per cent palladium-on-charcoal, to give compound I. The reaction of

III with ammonia leads to elimination, rather than to nucleophilic displacement.



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BIOCHEMISTRY

Micro-technique for Cell Electrophoresis

SEVERAL apparatuses have been described for measuring the electrical charge of small particles, such as cells¹⁻⁹. Due to the relatively large volume of the chambers used, and the minimum cell concentration necessary for adequate measurements, the total number of cells required for each measurement is rather large. When working with small samples, one sometimes finds it impossible to meet these requirements, and micromethods have therefore been developed. Theoretically, three different approaches are possible: to decrease the total volume of the chamber, to build the apparatus from several parts and fill only one of them with the cell suspension, or to introduce the sample to the measuring-place of a previously-filled chamber. The first of these possibilities is made complicated because of contamination by the electrodes. The second has been used by Seaman and Heard¹⁰, who built their apparatus from three parts. In their device only the microelectrophoresis tube is filled with the sample, while the electrode compartments remain 'cell-free'. This system handles samples of the order of 1 ml. or even less. The third possibility has recently been explored by Forrester *et al.*¹¹, who have been able to reduce the minimum suspension volume to 0.1–0.2 ml. (ref. 12). The small size and low cell numbers of the organ rudiments we have been investigating have forced us to use even smaller volumes. This led us to modify the apparatus described by Bangham *et al.*⁹.

The modification is illustrated in Fig. 1. It is based entirely on the apparatus described by Bangham *et al.* and produced for our laboratory by Rank Bros., Bottisham, Cambs. Therefore, only alterations to this apparatus will be described here. To the original U-tubing (G) two additional horizontal 'Pyrex' tubes 2 mm in internal diameter are fitted (D_1 – D_2), which are closed symmetrically with glass taps of vacuum quality (C_2 , C_4). The diameter of the drilled holes of these taps is also 2 mm. In addition, two vertical tubes closed by similar glass taps (C_1 , C_3) have been fitted to the lateral tubes in corresponding positions. The necks of the upper ends of the vertical tubes are 10 mm in diameter (B_1 , B_2). The lateral tubes penetrate the glass walls of the waterbath through drilled holes 50 mm in diameter, and to facilitate manipulation the left lateral tap is left outside the waterbath. A pair of rubber collars seal the holes in the glass wall against leakage while allowing the tube to be easily placed into the bath and to be moved vertically when calibrating the apparatus⁹. A mirror (M) has been placed under the microscope at a 45° angle in order to follow the pipetting of the sample.