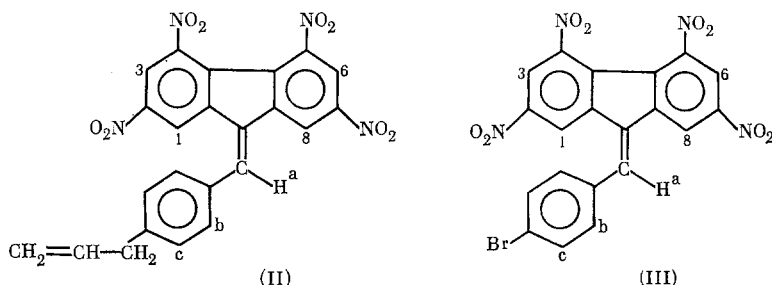


PREPARATION OF 2,4,5,7-TETRANITROFLUORENE
AND 9-ARYLIDENE-2,4,5,7-TETRANITROFLUORENES

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The nitration of fluorene by nitric acid ($d\ 1.51$) in concentrated sulfuric acid at 45-50°C for 5-6 h gives 2,4,5,7-tetranitrofluorene (I) as light brown crystals from benzene. The yield was 50%, mp 267-268°C (dec.) [1]. Found, %: C 44.66; H 1.69; N 15.63. Calculated, %: C 45.09; H 1.73; N 16.19. PMR spectrum (in CD_3CN , δ , ppm): 8.86, 8.85 (3H, 6H), 8.79, 8.78 (1H, 8H), 4.50 (9H). Mass spectrum (m/z): 346, 330, 300, 254, 238, 192. The reaction of (I) with 4-allylbenzaldehyde in DMF at 25°C for 2.5 h gives 9-L4-allylbenzylidene-2,4,5,7-tetranitrofluorene (II). PMR spectrum in CD_3CN : 9.10 (3H), 8.87 (6H), 8.71 (1H), 8.66 (8H), 8.53 (a), 7.61, 7.41 (b, c), $J_{b,c} = 8.04$ Hz; the system characteristic for the allylic fragment has signals at δ 5.96-6.16, 5.12-5.22, and 3.49-3.53 ppm. Mass spectrum (m/z): 474, 448, 306, 276. Product (III) was obtained by analogy. PMR spectrum in CD_3CN : 9.22 (3H), 9.22 (3H), 8.94 (6H), 8.82 (1H), 8.76 (8H), 8.59 (a), 7.66, 7.81 (b, c) ($J_{b,c} = 8.61$ Hz). Mass spectrum: 512, 482, 340, 310. The PMR spectra were taken on a Bruker WP-200 spectrometer and the mass spectra were taken on a Hitachi M-80A spectrometer.



LITERATURE CITED

1. Y. Abe, J. Chem. Soc. Jap. Chem. Ind. Chem., 1966 (1981).