removed periodically, denitrated and examined by paper chromatography as described above. The reaction was terminated after 96 hr., at which time no methyl D-glucoside could be detected in the products. Reactions at higher temperatures effected decomposition of the nitrate derivative.

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N,N'-Diethylene-*N''*-ethyl-*N''*-(1,3,4-thiadiazol-2-yl)phosphoramide

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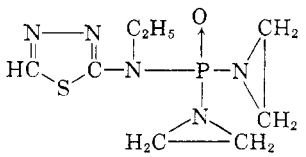
Carcinostatic activity has been reported for many derivatives of diethylenephosphoramide[1] and also for 2-ethylamino-1,3,4-thiadiazole.[2] It was therefore of interest to synthesize and test *N,N'* - diethylene - *N''* - ethyl - *N''* - (1,3,4 - thiadiazol - 2 - yl)phosphoramide (I), a potential "dual antagonist" incorporating these two active moieties



I

in one molecule. The product showed substantial activity against transplanted mouse tumors Sarcoma 180, 6C3HED lymphosarcoma, and C3H mammary adenocarcinoma by both oral and intraperitoneal administration. The synthesis is described below, and details of the testing will be reported elsewhere.[3] The compound is now undergoing clinical evaluation.

EXPERIMENTAL

N-Ethyl-*N*-(1,3,4-thiadiazol-2-yl)amidophosphoryl chloride was prepared by refluxing 16.4 g. (0.1 mole) of 2-ethylamino-1,3,4-thiadiazole hydrochloride[4] with 50 ml. of phosphorus oxychloride for 6 hr. and then removing excess phosphorus oxychloride by distillation under reduced pressure. The residual oil was washed with cold petroleum ether, (b.p. 30–60°), dried, and dissolved in 350 ml. of warm dry benzene. This solution was added slowly to a mixture of 9.5 g. (0.22 mole) of ethylenimine, 30.3 g. (0.3 mole) of triethylamine and 50 ml. of dry benzene at 10°. Agitation was continued for 2 hr. without cooling, after which the precipitated triethylamine hydrochloride was filtered off. The benzene was removed from the filtrate under reduced pressure, and the crude *N,N'*-diethylene-*N''*-ethyl-*N''*-(1,3,4-thiadiazol-2-yl)phosphoramide (19.7 g.) was purified by recrystallization from hexane; m.p. 95–96.5°.

Anal. Calcd. for $C_8H_{14}N_5OPS$: C, 37.1; H, 5.44; N, 27.0; S, 12.4. Found: C, 37.4; H, 5.67; N, 27.0; S, 12.6.

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The Pyrolysis of 2-Methylnaphthalene

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The wide occurrence of traces of benzo(g,h,i)-perylene in many petroleum products, such as waxes and solvents,[1] in which other six ring aromatic hydrocarbons are seldom found, suggested the possibility that this compound might be produced in the thermal procedures (*e.g.*, distillation) by which these products are manufactured. It is possible that benz(g,h,i)perylene is formed by condensation of smaller molecules, which process could be studied by pyrolysis of likely precursors. Pyrolysis of simple compounds has produced polycyclic aromatic hydrocarbons in small amounts.[2] Recently Badger and Kimber have studied the pyrolysis of tetralin[3] and of indene[4] which yielded, among other compounds, benzo(a)pyrene and benzo(j)fluoranthene.

The pyrolysis of 2-methylnaphthalene was studied, naphthalenes being common constituents of many petroleums. The vaporized methylnaphthalene was passed through a heated copper tube, and the material leaving the tube collected and analysed by chromatography.

Most of the 2-methylnaphthalene passed through the tube unchanged, even at 950°. The only product in amounts large enough for crystallization was 2,2'-binaphthyl. Some 1,2'-binaphthyl was present but could not be isolated and, like the other products, was identified by absorption spectroscopy. The identity of most of the products was confirmed by fluorescence spectroscopy. The calculated yields

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