TABLE I

MERCURIC NITRATE–PYRIDINE BASE ADDITION COMPOUNDS

(a) Desiccator dried; (b) vacuum dried; (c) nitric acid decomposition, reference 5; (d) hydrogen peroxide decomposition, reference 4

Pyridine base	M.p., °C. (a)	M.p., °C. (b)	Analyses (% Hg) (a) (c)	(a) (d)	(b) (c)	(b) (d)	Calcd. for 2 base/1Hg(NO_3)$_2$
Pyridine	234–236[e]	233–235	41.6	42.2	40.7	41.7	42.4
Quinoline	178 dec.[f]	179–181 dec.	34.8	35.1	35.0	35.2	34.6
β-Picoline	131–133 dec.	174–175 dec.	37.6	37.2	38.4	38.0	39.3
Lepidine	148 dec.	137 dec.	35.2	34.5	31.8	32.1	32.8
2,3-Lutidine	104–106 dec.	145	44.5	46.6	38.1	38.0	37.2[g]
α-Picoline	122–125 dec.	109–115 dec.	51.0	48.7	..	..	50.4[h]

[e] Pyridine nitrate, m.p. 118, sublimes during determination. Reported to decompose without melting; reference 2. [f] Reported, m.p. 183°; reference 3. [g] 1/1 Ratio; Hg. calcd. 46.5%. [h] Value calcd. for 1/2 ratio.

curic nitrate except for the 2,3-lutidine complex and the α-picoline complex for which the analyses agreed with a one to one ratio and a one to two ratio, respectively. After vacuum drying the analyses were in at least fair agreement with the values prior to vacuum drying except again for the 2,3-lutidine complex which analyzed for a two to one base to salt ratio. This change in ratio, involving a loss of mercury, can be explained as a loss of the volatile nitrate from the less stable one to one complex. The change in melting point on vacuum drying observed with this compound is consistent with the possibility of such a change. The α-picoline complex, which sublimed on vacuum drying, was not analyzed as a vacuum-dried sample. These analytical data do not require that these complexes contain water of crystallization.

Experimental

The mercuric nitrate reagent was prepared by suspending 216.6 g. (1 mole) of yellow mercuric oxide in 91.4 g. of concd. nitric acid and stirring until solution was complete. The addition compounds were precipitated by adding 0.1 mole of the base to 26.3 g. (0.1 mole) of the reagent with stirring and cooling over a five-minute period. The precipitated solid was collected on a filter and recrystallized from water (pyridine, β-picoline, quinoline, lepidine) or from ethanol (α-picoline, 2,3-lutidine). The solids were dried in a desiccator over anhydrous calcium chloride and potassium hydroxide or in drying pistol over phosphorus pentoxide at 2–6 mm. Melting points are uncorrected. Mercury analysis were made by procedures previously described.[4,5] Data are recorded in the Table.

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NEW COMPOUNDS

Tetraallylethylenediamine and Hexaallylethylenediammonium Dibromide

In the course of polymerization studies in this Laboratory,[1] tetraallylethylenediamine was prepared as an intermediate for the preparation of hexaallylethylenediammonium dibromide. The physical constants of these compounds have not been previously reported. Since polymerization studies of the quaternary ammonium salt as well as the properties of the polymer have been reported in a later paper,[2] it seems important that the preparation and properties of these compounds be recorded.

Tetraallylethylenediamine.—To a suspension of 67.2 g. of $NaHCO_3$ in 65 cc. of water was added 69 g. (0.71 mole) of diallylamine. With stirring, 66 g. (0.35 mole) of ethylene bromide was added dropwise. After the addition was complete, the mixture was heated under reflux for nine hr. After cooling, the sodium bromide was removed by filtration, and the filtrate saturated with NaOH. The amine layer was separated, dried over solid NaOH, and distilled; yield 32 g. (41%), b.p. 92° (3 mm.), d^{25}_{25} 0.8517, $n^{25}D$ 1.4702.

Anal. Calcd. for $C_{14}H_{24}N_2$: N, 12.71. Found: N, 12.62.

Hexaallylethylenediammonium Dibromide.—To 58 g. (0.26 mole) of tetraallylethylenediamine dissolved in 50 cc. of dry acetophenone, was added dropwise, with stirring, 63 g. (0.52 mole) of allyl bromide. The solution became warm on addition of the allyl bromide. Stirring was continued for four hr. after which time the salt began to precipitate. After an additional two hr., the salt was removed by filtration, washed several times with dry ether and dried; yield 108 g. (90%), m.p. 140°. After recrystallization from ethanol, the product had a melting point of 142°.

Anal. Calcd. for $C_{20}H_{34}N_2Br_2$: Br, 34.57. Found: Br, 34.28.

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Some Sulfonium and Selenonium Salts[1]

Continuing the search for additional compounds which cause damage to tumor cells *in vivo*,[2] salts have been prepared by reaction of methyl sulfide, methyl ethyl sulfide, ethyl sulfide, bis-(2-hydroxyethyl) sulfide, and ethyl selenide with *p*-fluorophenacyl chloride, *p*-phenylphenacyl bromide and β-naphthacyl bromide. In some cases the reaction was carried out in refluxing alcohol solution according to the method of Bost and Schultzer,[3] but in other cases best results were obtained by mixing the reactants and allowing them to stand at room temperature. Attempts to prepare sulfonium salts from bis-(2-cyanoethyl) sulfide were unsuccessful. The methods used are illustrated below and the properties of the products are listed in Table I. The salts were white or light buff solids which decomposed slowly on standing at room temperature and melted with decomposition on heating. The β-naphthacyl and *p*-phenylphenacyl salts were very slightly soluble in water (about 1 to 4 mg./ml.), while all the *p*-fluorophenacyl salts, except that of ethyl selenide, were more soluble.

Method I.—A solution of 7.25 g. of *p*-fluorophenacyl chloride in a slight excess of methyl sulfide was kept at room temperature for 1 day, then 6.8 g. (65%) of *p*-fluorophenacyl dimethyl sulfonium chloride was recovered by suction filtration and recrystallized from warm methanol by gradual

(1) G. B. Butler and R. L. Bunch, THIS JOURNAL, **71**, 3120 (1949).
(2) G. B. Butler, R. L. Bunch and F. L. Ingley, *ibid.*, **74**, 2543 (1952).

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(2) C. T. Bahner, *Texas Reports on Biology and Medicine*, **8**, 448 (1950); Henry A. Rutter, Jr., THIS JOURNAL, **73**, 5905 (1951).
(3) R. W. Bost and H. C. Schultzer, *ibid.*, **64**, 1165 (1942).