

The free amino-acids present in *Aspergillus niger* were determined under various mineral deficiencies by paper chromatography. The results, which are preliminary, are as follows:

Glutamic acid is considerably reduced when Fe, Zn, Cu and Mo are omitted but there is a tendency for it to increase in the absence of Mn. In the absence of Mo there is a marked reduction in amino-acid content and the only one detected was glutamic acid, thus coinciding with accumulation of nitrate N. Aminobutyric acid is increased when Fe and Mo are omitted respectively. Arginine and probably lysine occur in fungi deficient in Fe, Zn, Cu and Mn and are absent from those given full nutrient or those without Mo. The omission of Cu showed an increase in all amino-acids (except glutamic, which was slightly less) as compared with a full culture. Several amino-acids not found in fungi under other micronutrient deficiencies were observed, but their identification is as yet provisional.

Thus the free amino-acid content appears to vary under conditions of micronutrient deficiencies and more work is required to study these differential effects. An understanding of the role of the mineral elements in the physiology of the fungus is important to determine whether under all conditions it is specific for the 'test nutrient.'

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THE VAPOUR PRESSURE OF PARATHION AND RELATED COMPOUNDS

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In view of the widespread interest in insecticidal formulations based on parathion, the vapour pressures of the pure material and a number of related compounds likely to be encountered in the commercial product have been measured at various temperatures within the range 25–92° C. The materials examined include: (a) pure parathion, *O,O'*-diethyl *O-p*-nitrophenyl thiophosphate, (b) *S*-ethyl isomer of (a), *O,S*-diethyl *O-p*-nitrophenyl phosphate, (c) *S*-phenyl isomer of (a), *O,O'*-diethyl *S-p*-nitrophenyl phosphate, (d) commercial parathion, which contains mainly pure parathion together with small quantities of various related compounds, (e) *O,O',O''*-triethyl thiophosphate, (f) *O,O'*-diethyl *S*-ethyl phosphate, (g) *O*-ethyl *O,O'*-bis-*p*-nitrophenyl thiophosphate. Compounds (a) to (d) and (g) were examined by an effusion technique. Compounds (e) and (f) were examined in a liquid U-tube manometer. Details of the preparation of the materials are given. The vapour pressures have been shown to be low at ordinary temperatures (generally of the order of 10⁻⁴ mm. mercury, or lower, at 20° C.).

The data provided will be of use in assessing the toxic hazards of exposure to the vapour of parathion and related compounds, and the permanence of insecticidal deposits of the material.

Introduction

In the development of new insecticides a knowledge of the vapour pressure of the materials involved is important, among other reasons, in assessing the toxic hazards occurring in manufacture in an atmosphere which may contain the insecticide vapour and the permanence of the insecticidal deposit.

This paper deals solely with vapour-pressure determinations and is not concerned with any risks attendant upon physical contact with the liquid materials, nor with the absolute quantities of the vapours regarded as dangerous.

The material parathion (known originally in Germany as 'E605') has recently come into prominence as an acaricide and insecticide. This material is *O,O'*-diethyl *O-p*-nitrophenyl thiophosphate, but the commercially available product may contain the isomeric compounds, *O,S*-diethyl *O-p*-nitrophenyl phosphate and *O,O'*-diethyl *S-p*-nitrophenyl phosphate, and various related compounds such as *O,O',O''*-triethyl thiophosphate, *p*-nitrophenol, *O*-ethyl *O,O'*-bis-*p*-nitrophenyl thiophosphate and also some sodium thiophosphates as impurities.

Before any measurements of the vapour pressure of parathion were possible it was necessary to remove, as far as possible, the compounds quoted above. Although considerable information has been published on the preparation of the crude material,^{1, 2} on its properties and use under various conditions,^{3a, 3b} and on the analysis of small quantities of the

material,⁴ very little information is available on the vapour pressure of parathion. Since the material has a boiling point of 180° C. at a pressure below 1 mm. mercury, the vapour pressure is presumably very low at room temperature. It was originally^{3a} stated to have a vapour pressure corresponding to 20–25 mg./cu. m. at room temperature, but later evidence has reduced the figure to 10 mg./cu. m. and more recently to a still lower figure of 1 mg./cu. m. for pure parathion. Recently also, data have been quoted by the American Cyanamid Corporation at the 62nd Annual Meeting of the Florida State Horticultural Society which give the following figures for the vapour pressure of pure parathion:

Temperature, ° F.	Vapour pressure in microns of mercury				
80	0.038
100	0.11
120	0.37
140	1.04

There is thus scope for confirmation of these figures and for a measurement of the vapour pressure of the other materials for which no systematic information at all is available. The materials included within the scope of the investigation were: (a) pure parathion, *O,O'*-diethyl *O-p*-nitrophenyl thiophosphate, (b) *S*-ethyl isomer of (a), *O,S*-diethyl *O-p*-nitrophenyl phosphate, (c) *S*-phenyl isomer of (a), *O,O'*-diethyl *S-p*-nitrophenyl phosphate, (d) commercial parathion, containing mainly pure parathion with small amounts of various related compounds, (e) *O,O',O''*-triethyl thiophosphate, (f) *O,O'*-diethyl *S*-ethyl phosphate, (g) *O*-ethyl *O,O'*-bis-*p*-nitrophenyl thiophosphate.

Experimental

A. Preparation of pure parathion and related compounds

(i) *Isolation of pure parathion from crude commercial parathion*.—Since the distillation of crude parathion under vacuum at temperatures greater than 100° C. is a hazardous procedure which frequently results in violent decomposition, chromatographic separation appeared to offer a much better means of purification. The crude parathion, a commercial product, was first stripped of low-boiling impurities at 100° C. and approximately 0.1 mm. mercury pressure. 5 g. of the residue dissolved in 50 ml. benzene was repeatedly extracted with 2*N*-sodium hydroxide solution to remove the *p*-nitrophenol. After washing with water and drying with anhydrous sodium sulphate, the benzene solution was chromatographed on a column of fuller's earth, 50 cm. long and 2 cm. diameter, and then eluted with 150 ml. benzene. This eluate was concentrated to 50 ml., chromatographed on another column of fuller's earth and again eluted with 150 ml. benzene. This procedure was repeated on a further two columns of fuller's earth, the final eluate dried over anhydrous sodium sulphate and the benzene stripped off under vacuum. The residue was distilled from a 5-ml. flask using an air bath; the fraction boiling at a bath temperature of 120 to 140° C. at a pressure of 0.05 to 0.1 mm. mercury pressure was collected. This fraction was redistilled at 120 to 130° C. and a pressure of 0.05 to 0.1 mm. mercury. The yield amounted to 3 g. of very pale yellow liquid.

The microanalytical figures for the material were as follows: Found: O 41.1%; H 4.5%; N 5.1%; P 10.9%; S 11.2%. Required for *O,O*-diethyl *O-p*-nitrophenyl thiophosphate: O 41.2%; H 4.8%; N 4.8%; P 10.7%; S 11.0%.

(ii) *Preparation of pure synthetic parathion*.—This material was prepared by the condensation of sodium *p*-nitrophenate and diethoxythiophosphoryl chloride in aqueous solution at 100° C. after the method of Fletcher *et al.*¹⁰ The product was distilled slowly at 0.01 to 0.02 mm. mercury at a bath temperature not exceeding 115–120° C. in order to avoid any appreciable conversion from the sulphone form to the thiol isomer (see Schrader¹¹).

(iii) *O,S*-diethyl *O-p*-nitrophenyl phosphate. —This material was prepared from crude commercial parathion. 100 g. of the crude material was dissolved in 500 ml. ether and filtered. The filtrate was washed twice with 100-ml. lots of 0.1*N*-caustic soda solution and finally with two 100-ml. lots of distilled water. The ethereal solution was dried over anhydrous sodium sulphate and the ether removed by evacuation with a water pump. Triethyl thiophosphate was removed by evaporation at a bath temperature of 100° C. at 0.02 mm. mercury pressure. The residue was heated to a bath temperature of 180° C. and approximately 70 g. of the material allowed to distil.

The distillate was then heated at 130–140° C. for approximately 20 hr., after which time no sulphate ion could be detected after oxidation of a sample with 55% nitric acid.

On re-distillation of this material in small (10-g.) batches, the product was isolated as a yellow oil which distilled at a bath temperature of 160–170° C. at 0.02 mm. of mercury pressure.

(iv) *O,O'*-diethyl *S-p*-nitrophenyl phosphate. —This material was supplied by Albright and Wilson Ltd. It was a pale-yellow, low-melting-point solid, and was used as received.

(v) *O,O',O''*-triethyl thiophosphate. —This material was prepared on a molar scale in 80% of the theoretical yield following the procedures of Chevrier¹² and Pistschimuka,¹³ by the action of sodium ethoxide on thiophosphoryl trichloride. The ester was isolated as a colourless liquid having a boiling point of 79–82° C. at 10 mm. mercury pressure. For the vapour-pressure measurements, this ester was twice re-distilled through a vacuum-jacketed Vigreux-type column and finally a fraction was collected having a boiling point of 80–81° C. at 10 mm. mercury pressure and a refractive index, $n_D^{19} = 1.4510$.

(vi) *O,O'*-diethyl *S*-ethyl phosphate. —This was prepared by the heat treatment of *O,O',O''*-triethyl thiophosphate after the technique of Schrader¹¹ and Emmett and Jones.¹⁴

100 g. of the *O,O',O''*-triethyl thiophosphate was heated at 140–150° C. Small samples were withdrawn with a pipette at intervals and oxidized with 55% aqueous nitric acid. When the oxidized material no longer gave a test for sulphate ion with barium chloride solution, isomerization from the sulphone to the thiol form was considered to be complete; this state was usually attained after approximately 24 hours' heating. The material was then fractionated through a normal column and the fraction, weighing 40 g., coming over between 124° and 130° C. at 15 mm. pressure was collected.

Two further fractionations gave a final product of boiling point 113–115° C. at 10 mm. mercury pressure and refractive index, $n_D^{19} = 1.4574$. The final yield of 20 g. was the material used for the vapour pressure determinations.

An attempt was also made to synthesize the ester by the reaction of diethoxyphosphoryl chloride with the sodium derivative of ethyl mercaptan in toluene at 100–110° C.; only a poor yield of the desired material was obtained, and the method was not investigated further.

(vii) *O*-ethyl *O,O'*-bis-*p*-nitrophenyl thiophosphate. —31 g. of *p*-nitrophenol and 9 g. sodium hydroxide were dissolved in

75 ml. water and heated to 95–100° C. 20 g. of ethoxythiophosphoryl dichloride was added drop-wise with stirring over a period of 1 hr. Heating was continued for a further 1½ hr. The mixture was then cooled to room temperature and the pale-yellow crystalline material was filtered off. A yield of 21 g., 50% of the theoretical, of m.p. 126–131° C. was obtained. This material was recrystallized from a large volume of methylated spirits and the final product has the following properties :

Melting point	129–130° C.
Colour	Very pale yellow
Molecular weight (cryoscopically in benzene)	398 (theoretical for C ₁₄ H ₁₃ O ₇ N ₂ PS = 384)

Microanalysis

Found	N 7.6% ; S 8.4%.
C ₁₄ H ₁₃ O ₇ N ₂ PS requires	N 7.3% ; S 8.3%.

B. Vapour-pressure measurements

The technique of measurement of the vapour pressure of compounds (a) to (d) and (g) (see p. 345) was that involving the effusion method recently used by one of us⁵ for *cis*- and *trans*-azobenzene. The method was originally employed by Knudsen⁶ and later developed by Egerton⁷ for metals ; more recently it has been used in a modified form for DDT and other slightly volatile insecticidal materials by Balson.⁸

The material was heated for a known time at a constant temperature in a small copper vessel having a cylindrical orifice of known dimensions through which the vapour could effuse as a molecular beam into a highly evacuated space. The vapour pressure, *p*, of the substance expressed in millimetres of mercury, is given by the formula

$$p = \frac{g}{W.a.t.} \sqrt{\frac{M}{T}} 5.83 \times 10^{-2}$$

where *g* = weight (g.) of material effused through the orifice, estimated by the loss in weight of the container and substance

a = cross-sectional area of orifice, sq. cm.

t = time of duration of experiment, sec.

T = mean absolute temperature of determination measured by copper-constantan thermocouple soldered to bottom of copper vessel

M = molecular weight of substance under investigation

W = probability of effusion through the orifice for a given molecule ; the values of *W* for orifices of various length/cross-sectional area ratios have been calculated by Clausing.⁹

The experimental results were examined by least-square methods to give the best straight line connecting log₁₀ *p* with 1/*T*. The slope of this line gives the latent heat of evaporation.

The technique adopted for materials (e) and (f) (see p. 345), which are liquid in the temperature-range of interest, was that in which an all-glass U-tube manometer was used, having one limb closed and the other highly evacuated by a high-speed pumping system ; the whole was maintained at a series of steady known temperatures by immersion in water, and the material itself acted as its own manometric fluid ; the hydrostatic head of pressure was measured with a travelling microscope. As it was necessary to know the density of the

materials at the temperatures of the investigation to convert this hydrostatic head to millimetres of mercury pressure, the density had to be determined for each material at several temperatures by normal methods. The experimental values of the vapour pressure were treated statistically in the same way as those obtained using the effusion technique.

The experimental vapour-pressure figures, with the best straight-line graphs, are shown in Figs. 1, 2 and 3.

The only figure obtained for *O*-ethyl *O*,*O*'-bis-*p*-nitrophenyl thiophosphate was at 88.5° C., where the vapour pressure was below 10⁻⁶ mm. mercury.

The results of the density determinations are shown in Table I.

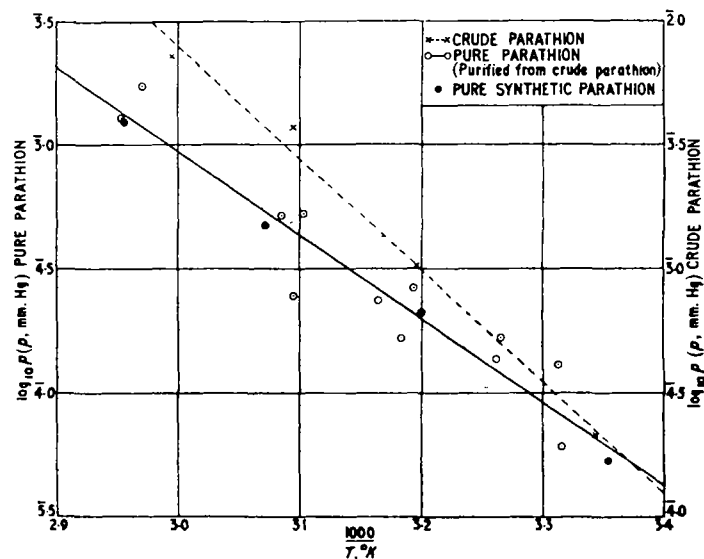


FIG. 1.—Vapour pressure of pure and crude parathion (using effusion technique)

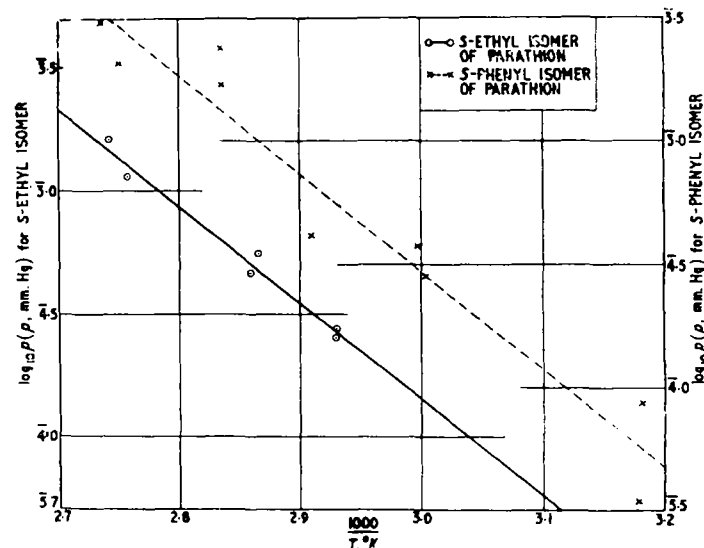


FIG. 2.—Vapour pressure of S-ethyl and S-phenyl isomers of parathion (by effusion technique)

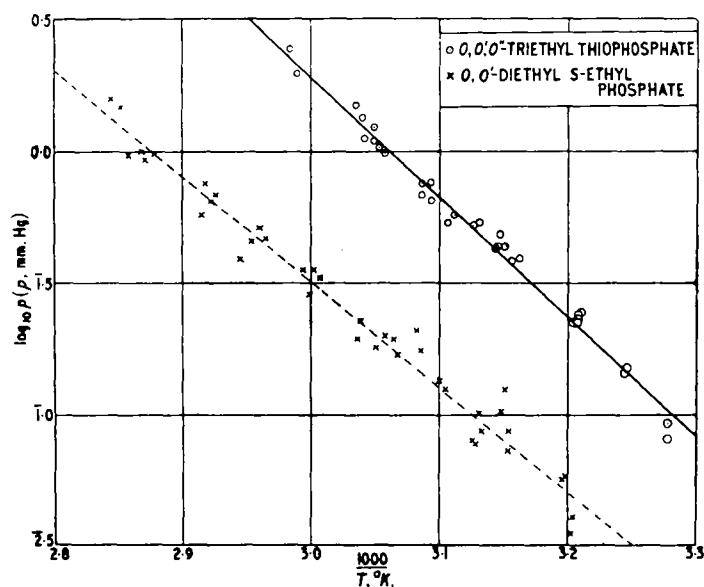


FIG. 3.—Vapour pressure of triethyl thiophosphates (by manometric technique)

Table I

Temperature, °C.	Density of material, g./c.c.	
	O,O',O''-triethyl thiophosphate	O,O'-diethyl S-ethyl phosphate
31.3	1.0783 ₄	1.0957 ₅
44.8	1.0650 ₆	1.0833 ₄
60.0	1.0491 ₈	1.0681 ₉

These two sets of points each lie approximately on straight lines, and it was considered that linear interpolations between each set gave the density sufficiently accurately for the present purposes.

Discussion

The experimental results, expressed in the form of 'best' straight lines having the equation

$$\log_{10} p = -\frac{A}{T} + B$$

where p = vapour pressure in mm. mercury
 T = absolute temperature of determination

are summarized in Table II, and were obtained in the usual way by least-square methods.

In order that the significance of these results might be more easily appreciated, the vapour pressures calculated for 20°C., 40°C., 60°C., 80°C. and 100°C. are given in mm. mercury for the various compounds in Table III.

The figures given for the crude material assume the molecular weight of the vapour to be same as for the pure compound, which is not strictly true; nevertheless they are in good accord with those obtained for the same material by an air-streaming method. The figures obtained were as follows:

Temperature, °C.	Amount of material carried over in 1 hr. by an air stream of 16 l./min.	
	Experimental, mg.	Calculated from effusion data, mg.
20	1	1.7
50	26	41.4

The figures for pure parathion are in agreement with that obtained elsewhere where a content of 1 mg. for 1 cu. m. saturated air was quoted; the figure calculated from our data is 0.7 mg. at 20°C. Our figures for pure parathion also appear to be in good accord with the data recently published by the American Cyanamid Corporation (see p. 345).

Temperature, °C.	Vapour pressure, mm. mercury	
	American data	Present work
26.8	3.8×10^{-5}	6.95×10^{-5}
37.8	1.1×10^{-4}	1.75×10^{-4}
49.0	3.7×10^{-4}	4.16×10^{-4}
60.0	1.04×10^{-3}	0.93×10^{-3}

The coefficient of variation, deduced from the least-squares plot, of an individual determination of the vapour pressure, is of the order of ± 10 to 15% . With the effusion data, this large scatter is due to the smallness of the quantity of the effusing material obtained in a convenient time. The loss in weight of the container, usually about 0.5 to 1.5 mg., could not readily be measured to better than ± 0.1 mg. because of the amount of manipulation necessary.

With the manometric data, the chief sources of error are in the control of temperature, the small hydrostatic heads to be measured in some cases and uncertainty in the position of the menisci due to vibration.

The data obtained in the present work on the two triethyl thiophosphates are in reasonable accord with the fragmentary density and boiling-point data quoted in the most recent issues of 'Beilstein,' namely:

(a) O,O',O''-triethyl thiophosphate

$$d_{40}^{20} = 1.0944$$

$$d_{40}^{20} = 1.1132$$

Boiling point at 20 mm. mercury 106°C.

" " at 12 mm. " 95.5°C.

(b) O,O'-diethyl S-ethyl phosphate

$$d_{40}^{20} = 1.1245$$

Boiling point at 16 mm. mercury 120°C.

" " at 20 mm. " 122°C.

Table II

Material	Temperature range investigated, °C.	A	B	Latent heat of vaporization, g.-cal./g.	Coefficient of variation of a determination of vapour pressure, %
(a) Pure parathion (O,O'-diethyl O-p-nitrophenyl thiophosphate)	25.2-65.5	3395	7.161	53.3	± 13.4
(b) O,S-diethyl O-p-nitrophenyl phosphate	59.2-91.5	3924	7.925	61.6	± 11.1
(c) O,O'-diethyl S-p-nitrophenyl phosphate	41.4-92.6	3966	8.370	62.3	± 15.0
(d) Crude parathion	26.1-60.8	4537	11.517	—	—
(e) O,O',O''-triethyl thiophosphate	32.0-62.0	4570	14.001	105.4	± 10.3
(f) O,O'-diethyl S-ethyl phosphate	39.1-78.4	3985	11.463	91.9	± 17.9

Table III

Material (see Table II)	Vapour pressures calculated from best straight lines, mm. Hg				
	20° C.	40° C.	60° C.	80° C.	100° C.
(a)	3.78×10^{-5}	2.08×10^{-4}	9.31×10^{-4}	3.52×10^{-3}	1.15×10^{-2}
(b)	3.44×10^{-6}	2.46×10^{-5}	1.39×10^{-4}	6.49×10^{-4}	2.56×10^{-3}
(c)	6.86×10^{-6}	5.02×10^{-5}	2.90×10^{-4}	1.37×10^{-3}	5.49×10^{-3}
(d)	1.09×10^{-4}	1.06×10^{-3}	7.87×10^{-3}	4.65×10^{-2}	2.28×10^{-1}
(e)	2.56×10^{-2}	2.53×10^{-1}	1.91	11.44	56.62
(f)	7.42×10^{-3}	5.48×10^{-2}	3.19×10^{-1}	1.52	6.12

It is interesting to observe that, so far as vapour pressure is concerned, *O,O'*-diethyl *S*-ethyl phosphate stands in the same relation to *O,O'O''*-triethyl thiophosphate as the *S*-ethyl isomer of parathion does to parathion itself.

It is considered that the data presented are adequate to meet technical requirements. It will be observed that in all cases, the vapour-pressure measurements are in accord with the distillation data given in the section on the preparation of the materials.

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Journal of the Society of Chemical Industry

The following papers are appearing in the November, 1950, issue of the *Journal of the Society of Chemical Industry*.

DESIGN OF PACKED DISTILLATION COLUMNS. I.—PRESSURE DROP THROUGH PACKED DISTILLATION COLUMNS. By G. H. G. Hands, F. R. Whitt and K. S. Gregory.	THE EFFECT ON WATER VAPOUR PRESSURE OF SUPERIMPOSED AIR PRESSURE. By T. J. Webster.
THE PROXIMATE ANALYSIS OF BENZALDEHYDE MONO-NITRATION PRODUCTS. By W. Davey and J. R. Gwilt.	SUPERFINE GRINDING OF COKE AND OTHER MATERIAL. By R. A. Mott.
MANUFACTURE OF ALGAL CHEMICALS. I.—PRODUCTION OF ALGINATES FROM BROWN MARINE ALGAE. By L. A. Bashford, R. S. Thomas and F. N. Woodward.	THE CHLORINATION OF α -CHLOROETHYLBENZENE. I. By E. Galitzenstein (the late) and C. Woolf.