J. Chem. Eng. Data **1982**, *27*, 34-38

Table III. Parameters and the Root Mean Square Deviations in Relative Pressures

	273.15 K			293.15 K		
	$w_{ij}{}^a$	$w_{ji}{}^a$	rms[b]	$w_{ij}{}^a$	$w_{ji}{}^a$	rms[b]
propane–propylene	−0.0187	0.0830	0.0015	0.0057	0.0458	0.0011
propane–tetralin	0.7742	0.0968	0.0095	0.7434	0.0976	0.0082
propylene–tetralin	0.7427	−0.1773	0.0069	0.7761	−0.2828	0.0085
propane–propylene–tetralin			0.0035			0.0070

[a] First component is *i*. [b] rms = $\{\Sigma_{i=1}{}^K[(P_{\text{calcd}} - P_{\text{exptl}})/P_{\text{exptl}}]_i{}^2/K\}^{1/2}$.

K	number of data
N	number of component
P	total pressure, kPa
$P_i{}^s$	vapor pressure of pure component, kPa
R	gas constant, J mol^{-1} K^{-1}
T	absolute temperature, K
v	molar volume of liquid, m^3 mol^{-1}
w_{ij}	adjustable parameter (w_{ii} = 0)
x	liquid mole fraction
y	vapor mole fraction

Greek Letters

γ	activity coefficient
ϕ	fugacity coefficient

Subscripts

1	propane
2	propylene
3	tetralin
i	component *i*

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Solubility of Urea in Ammonium Polyphosphate Solutions at 0 and 25 °C

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The six-component system $CO(NH_2)_2$–NH_3–H_3PO_4–$H_4P_2O_7$–$H_5P_3O_{10}$–H_2O, along with the subsystem $CO(NH_2)_2$–NH_3–H_3PO_4–$H_4P_2O_7$–H_2O, was studied at 0 and 25 °C to determine the solubility isotherms in the pH range 5–7. All invariant solutions were identified at both temperatures. In the two systems, those solutions containing monoammonium and diammonium orthophosphates in equilibrium had the highest total plant food.

This study of the solubility of urea in the presence of the three basic linear ammonium phosphates was made to complement previous solubility studies of the same phosphates (*1–4*). Determinations of the composition of solutions in the system urea–ammonia–orthophosphoric acid–pyrophosphoric acid–tripolyphosphoric acid–water in the pH range 5.2–7.2 were made at 0 and 25 °C, as well as the subsystem urea–ammonia–orthophosphoric acid–pyrophosphoric acid–water in the pH range 4.4–7.2 at 0 and 25 °C.

The urea and monoammonium and diammonium orthophosphates were reagent grade. Triammonium and tetraammonium pyrophosphates were crystallized from a product of ammoniation of 80% P_2O_5 electric-furnace superphosphoric acid. Pentaammonium tripolyphosphate was prepared from the sodium salt by using cation-exchange resin (Amberlite IR-120 H^+ form) to produce a tripolyphosphoric acid solution and then ammoniating to pH 7.8 before crystallizing the salt by the addition of methyl alcohol to the solution.

Two stock solutions saturated with monoammonium or diammonium orthophosphate were prepared at ambient temperature. Depending upon the desired pH (*5*) for the complex, a portion of one of the stock solutions whose saturating salt is stable at that pH was saturated with the ammonium pyrophosphate that is stable (*2*, *4*) at the desired pH. For the subsystem these ammonium orthophosphate–pyrophosphate mixtures were adjusted to the desired pH with gaseous ammonia or orthophosphoric acid. Urea was added, and the complexes were placed either in a cold room at 0 ± 0.5 °C with periodic manual agitation or in a water bath at 25 ± 0.2 °C and agitated at 4 rpm. This procedure was the same for the main system, except that the orthophosphate–pyrophosphate solutions were saturated with pentaammonium tripolyphosphate before adjusting the pH and saturating with urea.

The approach to equilibrium was followed by petrographic examination of the solid phase of the complexes. When one

Table I. System $CO(NH_2)_2$-NH_3-H_3PO_4-$H_4P_2O_7$-H_2O

	liquid phase						
	composition, %				distribution of P_2O_5, %		
	nitrogen						
pH	total	NH_3	urea	P_2O_5	ortho	pyro	solid phase[a]
				0 °C			
7.25	15.8	7.2	8.6	20.36	49.2	50.8	B, E, F
7.15	15.7	7.4	8.3	21.58	44.9	55.1	B, E, F
7.06	15.4	7.6	7.8	22.23	43.1	56.9	B, E, F
6.77	15.3	8.4	6.9	25.57	41.1	58.9	B, E, F
6.59	15.1	9.3	5.8	29.11	35.9	64.1	B, D, E, F
6.22	15.1	9.0	6.1	29.92	47.7	52.3	B, D, F
6.08	14.9	9.1	5.8	31.07	50.7	49.3	A, B, D, F
5.95	14.9	8.4	6.5	29.73	45.2	54.8	A, D, F
5.78	14.9	8.4	6.5	29.67	44.8	55.2	A, D, F
5.67	15.0	7.6	7.4	28.60	36.9	63.1	A, D, F
5.09	15.0	7.5	7.5	29.56	25.4	74.6	A, C, D, F
5.03	15.5	7.0	8.5	27.92	26.6	73.4	A, C, F
5.01	15.5	6.7	8.8	27.27	28.2	71.8	A, C, F
4.65	16.6	4.5	12.1	20.00	34.5	65.5	A, C, F
4.39	16.7	4.3	12.4	19.30	36.6	63.4	A, C, F
				25 °C			
7.12	21.3	4.7	16.6	14.25	47.6	52.4	B, E, F
6.84	19.9	6.1	13.8	19.21	46.8	53.2	B, E, F
6.34	18.1	8.4	9.7	28.16	41.5	58.5	B, E, F
6.15	17.7	9.0	8.7	30.87	39.9	60.1	B, D, E, F
5.85	17.0	9.3	7.7	32.75	46.3	53.7	B, D, F
5.78	17.2	9.5	7.7	33.71	47.9	52.1	A, B, D, F
5.46	17.7	8.6	9.1	31.88	37.4	62.6	A, D, F
5.37	17.8	8.4	9.4	31.59	35.7	64.3	A, D, F
5.27	17.8	8.1	9.7	31.68	31.9	68.1	A, D, F
5.25	17.9	8.1	9.8	32.07	31.3	68.7	A, D, F
5.12	17.8	8.2	9.6	32.58	27.5	72.5	A, C, D, F
5.01	18.6	6.9	11.7	28.58	30.3	69.7	A, C, F
4.81	20.4	5.1	15.3	22.38	34.3	65.7	A, C, F
4.60	21.1	4.3	16.8	19.95	36.0	64.0	A, C, F
4.39	21.7	3.7	18.0	18.32	38.7	61.3	A, C, F

[a] A = $NH_4H_2PO_4$, B = $(NH_4)_2HPO_4$, C = $(NH_4)_2H_2P_2O_7$, D = $(NH_4)_3HP_2O_7 \cdot H_2O$, E = $(NH_4)_4P_2O_7 \cdot H_2O$, F = $CO(NH_2)_2$.

or more of the saturating salts were absent from the solid phase, a small amount was added and equilibrium continued. When two successive examinations (usually 1 week apart) of the solid phase showed that there was no change, samples of the liquid phase were taken for determination of pH, composition, and distribution of phosphate species. The pH was determined with a commercial meter and glass electrode, phosphorus was determined gravimetrically as quinolinium molybdophosphate, nitrogen was determined by distillation of ammonia after decomposing with Devarda's alloy, and the distribution of phosphate species was determined by one-dimensional paper chromatography (*6*, *7*).

Results

Subsystem $CO(NH_2)_2$-NH_3-H_3PO_4-$H_4P_2O_7$-H_2O. The time required for establishing equilibrium ranged from 7 to 28 days. The results are summarized in Table I and plotted in Figures 1–3. As shown in Figure 1, each isotherm consists of four branches over the pH range covered. As the ratio (or pH) NH_3-N:P_2O_5 increased, the compositions of the saturating phases of the branches were $CO(NH_2)_2$–$NH_4H_2PO_4$–$(NH_4)_2H_2P_2O_7$, $CO(NH_2)_2$–$NH_4H_2PO_4$–$(NH_4)_3HP_2O_7 \cdot H_2O$, $CO(NH_2)_2$–$(NH_4)_2HPO_4$–$(NH_4)_3HP_2O_7 \cdot H_2O$, and $CO(NH_2)_2$–$(NH_4)_2HPO_4$–$(NH_4)_4P_2O_7 \cdot H_2O$. Three invariant solutions were determined at each temperature. The invariant solution containing monoammonium and diammonium orthophosphates in equilibrium had the highest total plant nutrient value of all solutions in the pH range 4.4–7.2.

It is shown in Figure 1 that those solutions above the ratio NH_3-N:P_2O_5 of 0.305 (pH >6.35) contained less total plant food at 25 °C than at 0 °C. The composition of the saturated solutions (Figure 2) shows a minimum for urea when the P_2O_5 is greatest and a rapid decrease in the P_2O_5 content when the urea concentration rises. The urea concentration rises more rapidly at 25 °C than at 0 °C as the pH increases or decreases from the minimum, and this salting-out effect explains the higher total plant-food content at 0 °C than at 25 °C at high pH.

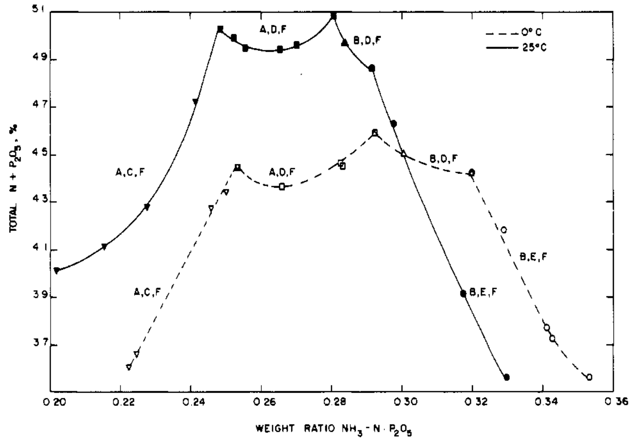

Figure 1. Solubility in the system $CO(NH_2)_2$–NH_3–H_3PO_4–$H_4P_2O_7$–H_2O at 0 and 25 °C. A = $NH_4H_2PO_4$, B = $(NH_4)_2HPO_4$, C = $(NH_4)_2H_2P_2O_7$, D = $(NH_4)_3HP_2O_7 \cdot H_2O$, E = $(NH_4)_4P_2O_7 \cdot H_2O$, F = $CO(NH_2)_2$.

Some plant-food ratios are shown in Figure 3 as a function of pH. As with the solubility isotherms, these ratios comprise four branches. At the invariant pH of monoammonium and diammonium orthophosphates, the ratio NH_3-N:P_2O_5 is a maximum at 25 °C; and at 0 °C, this invariant pH and that of

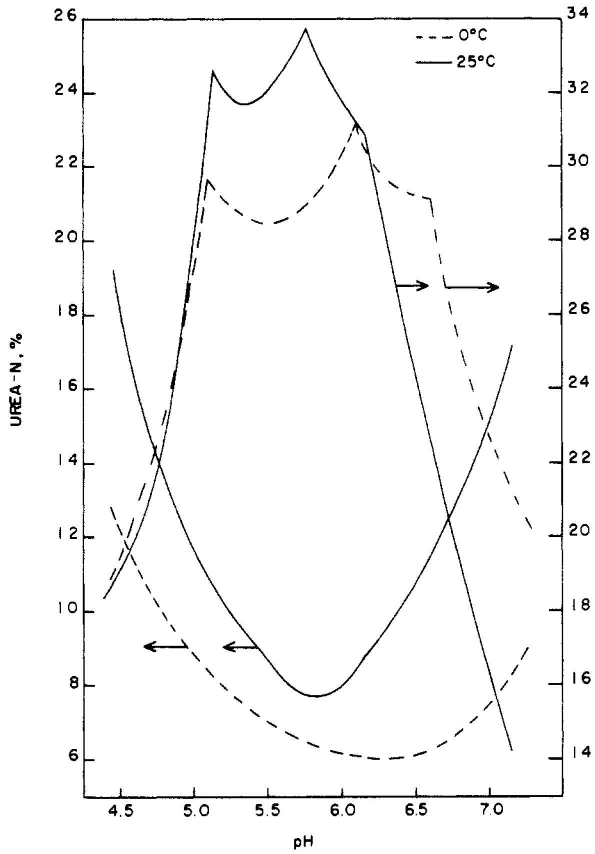

Figure 2. Relationship of urea and P_2O_5 concentrations with pH in the system urea-NH_3-H_3PO_4-$H_4P_2O_7$-H_2O at 0 and 25 °C.

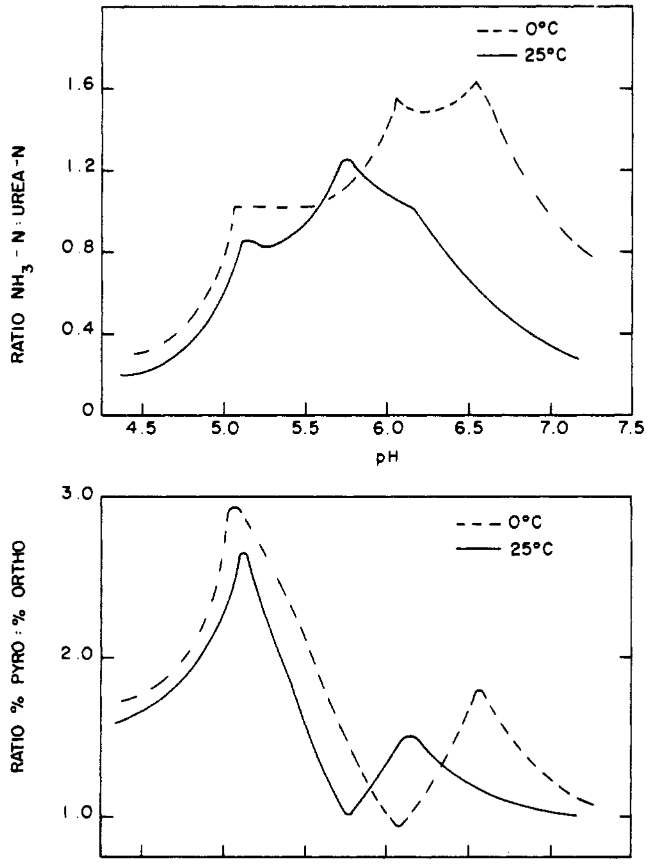

Figure 3. Distributions of N and P_2O_5 with pH in the system $CO(NH_2)_2$-NH_3-H_3PO_4-$H_4P_2O_7$-H_2O at 0 and 25 °C.

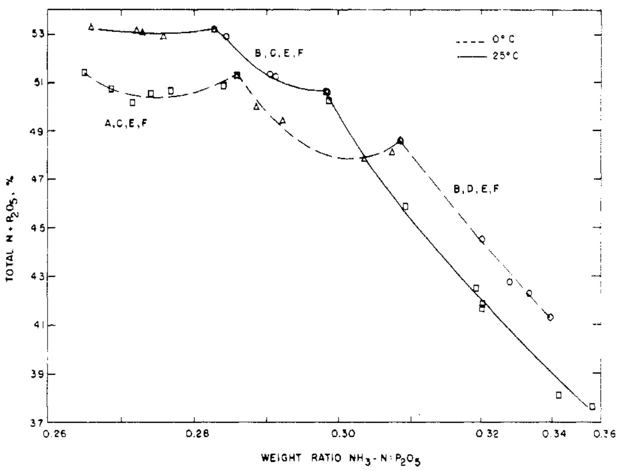

Figure 4. Solubility in the system $CO(NH_2)_2$-NH_3-H_3PO_4-$H_4P_2O_7$-$H_5P_3O_{10}$-H_2O at 0 and 25 °C. A = $NH_4H_2PO_4$, B = $(NH_4)_2HPO_4$, C = $(NH_4)_3HP_2O_7 \cdot H_2O$, D = $(NH_4)_4P_2O_7 \cdot H_2O$, E = $(NH_4)_5P_3O_{10} \cdot 2H_2O$, F = $CO(NH_2)_2$.

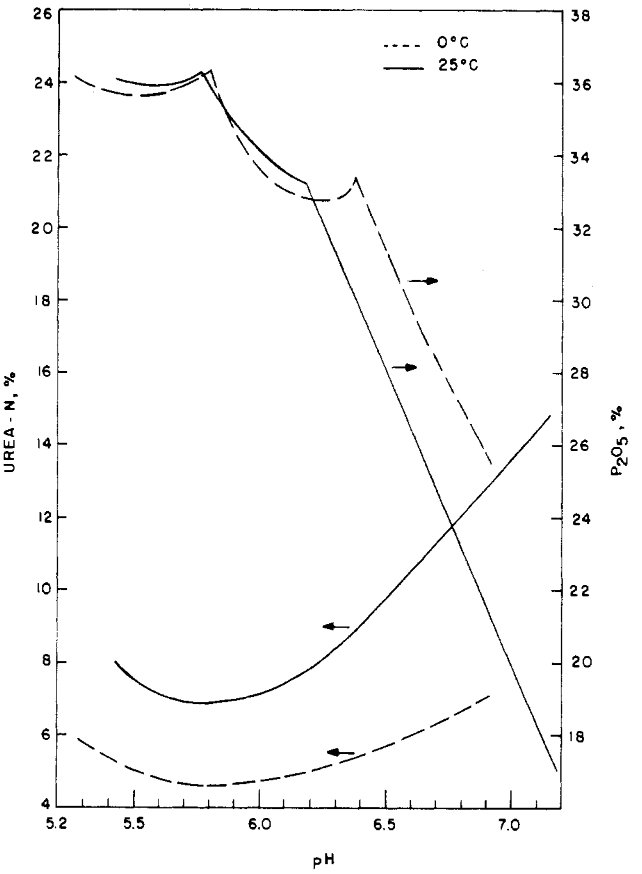

Figure 5. Concentrations of urea and P_2O_5 with pH in the system urea-NH_3-H_3PO_4-$H_4P_2O_7$-$H_5P_3O_{10}$-H_2O at 0 and 25 °C.

triammonium and tetraammonium pyrophosphates give ratios NH_3-N:urea-N that are approximately equal. The phosphorus ration pyro:ortho is ~1 at the invariant pH of monoammonium and diammonium orthophosphates and then approaches 1 again as the pH increases to 7.2.

The pH of the saturated solutions is related to the weight ratio NH_3-N:P_2O_5 (R) at 0 °C by

$$\mathrm{pH} = -0.26 + 21.49R \qquad (1)$$

with a correlation coefficient of 0.995 and a prediction standard deviation of 0.10 pH unit, and at 25 °C by

$$\mathrm{pH} = -0.18 + 21.56R \qquad (2)$$

Table II. System $CO(NH_2)_2$-NH_3-H_3PO_4-$H_4P_2O_7$-$H_5P_3O_{10}$-H_2O

	liquid phase							
	composition, %							
	nitrogen				distribution of P_2O_5, %			
pH	total	NH_3	urea	P_2O_5	ortho	pyro	tripoly	solid phase[a]
				0 °C				
6.90	15.6	8.5	7.1	25.78	33	40	27	B, D, E, F
6.84	15.4	8.8	6.6	26.93	33	41	26	B, D, E, F
6.71	15.3	8.9	6.4	27.47	32	44	24	B, D, E, F
6.61	15.2	9.3	5.9	28.91	32	46	22	B, D, E, F
6.59	15.2	9.4	5.8	29.36	31	48	21	B, D, E, F
6.37	15.3	10.3	5.0	33.39	32	52	16	B, C, D, E, F
6.28	15.3	10.1	5.2	32.80	33	49	18	B, C, E, F
6.15	15.0	10.0	5.0	32.94	38	44	18	B, C, E, F
5.90	14.9	10.1	4.8	34.52	39	42	19	B, C, E, F
5.83	15.1	10.1	5.0	34.98	40	39	21	B, C, E, F
5.80	15.0	10.4	4.6	36.31	46	39	15	A, B, C, E, F
5.78	15.1	10.2	4.9	35.88	42	38	20	A, C, E, F
5.53	14.6	10.0	4.6	36.09	35	41	24	A, C, E, F
5.46	14.8	9.8	5.0	35.72	28	40	32	A, C, E, F
5.44	15.2	9.5	5.7	34.96	28	40	32	A, C, E, F
5.38	15.0	9.6	5.4	35.74	29	40	31	A, C, E, F
5.26	15.1	9.6	5.5	36.14	26	42	32	A, C, E, F
				25 °C				
7.24	20.7	5.7	15.0	16.99	39	31	30	B, D, E, F
7.12	20.6	5.8	14.8	17.54	39	32	29	B, D, E, F
6.82	19.4	7.2	12.2	22.46	37	39	24	B, D, E, F
6.80	19.3	7.2	12.1	22.45	37	39	24	B, D, E, F
6.71	19.4	7.4	12.0	23.16	37	39	24	B, D, E, F
6.52	18.4	8.5	9.9	27.47	36	44	20	B, D, E, F
6.34	17.8	9.7	8.1	32.44	34	48	18	B, D, E, F
6.18	17.5	9.9	7.6	33.12	35	50	15	B, C, D, E, F
6.03	17.3	9.9	7.4	33.96	37	47	16	B, C, E, F
5.95	17.3	9.9	7.4	34.05	37	47	16	B, C, E, F
5.82	17.1	10.2	6.9	35.84	40	43	17	B, C, E, F
5.78	17.0	10.2	6.8	36.22	40	43	17	B, C, E, F
5.76	17.1	10.3	6.8	36.24	42	42	16	A, B, C, E, F
5.62	17.1	9.9	7.2	35.84	35	44	21	A, C, E, F
5.55	17.2	9.8	7.4	35.88	33	44	23	A, C, E, F
5.52	17.2	9.8	7.4	35.99	33	44	23	A, C, E, F
5.43	17.2	9.6	7.6	36.09	29	44	27	A, C, E, F

[a] A = $NH_4H_2PO_4$, B = $(NH_4)_2HPO_4$, C = $(NH_4)_3HP_2O_7{\cdot}H_2O$, D = $(NH_4)_4P_2O_7{\cdot}H_2O$, E = $(NH_4)_5P_3O_{10}{\cdot}2H_2O$, F = $CO(NH_2)_2$.

with a correlation coefficient of 0.986 and a prediction standard deviation of 0.14 pH unit.

System $CO(NH_2)_2$–NH_3–H_3PO_4–$H_4P_2O_7$–$H_5P_3O_{10}$–H_2O. Equilibrium was established in a minimum of 7 days. The results are summarized in Table II and in Figures 4–6. The coordinates of Figure 4 were chosen to show the total plant food (total N + P_2O_5) over the pH range (NH_3-N:P_2O_5) studied, which is normal for the production of plant-food solutions. In the pH range studied, the 0 and 25 °C isotherms comprise three branches representing solutions saturated with $NH_4H_2PO_4$, $(NH_4)_3HP_2O_7{\cdot}H_2O$, $(NH_4)_5P_3O_{10}{\cdot}H_2O$, and $CO(NH_2)_2$; $(NH_4)_2HPO_4$, $(NH_4)_3HP_2O_7{\cdot}H_2O$, $(NH_4)_5P_3O_{10}{\cdot}H_2O$, and $CO(NH_2)_2$; and $(NH_4)_2HPO_4$, $(NH_4)_4P_2O_7$, $(NH_4)_5P_3O_{10}{\cdot}H_2O$, and $CO(NH_2)_2$. Two invariant solutions were observed at both temperatures. The invariant solution containing monoammonium and diammonium orthophosphates has the highest total plant food in the pH range studied at 0 and 25 °C, although the plant-food content changes only slightly as the pH decreases from the invariant solution.

Figure 5 shows the solubility of urea and P_2O_5 at each temperature as a function of pH. The urea is least soluble at pH ~5.8, which coincides with the maximum P_2O_5 content. The increase in urea concentration at 25 °C as the pH increases from 5.8 is greater than that at 0 °C. The salting out of P_2O_5 salts by the increased amounts of urea results in a greater plant-food solubility at 0 °C than at 25 °C as also was observed in the subsystem.

Figure 6 shows the phosphate distribution over the pH range studied for both temperatures. As expected, the orthophosphate and pyrophosphate were greatest in those solutions that contained two salts of the species in equilibrium. The fraction of phosphate as tripolyphosphate was always greater at 0 °C than at 25 °C; the same was true for orthophosphate above pH 6.0 and for pyrophosphate below pH 6.25. When compared to the urea-free system (*1–4*), the addition of urea suppressed the solubility of P_2O_5 over the pH range studied at

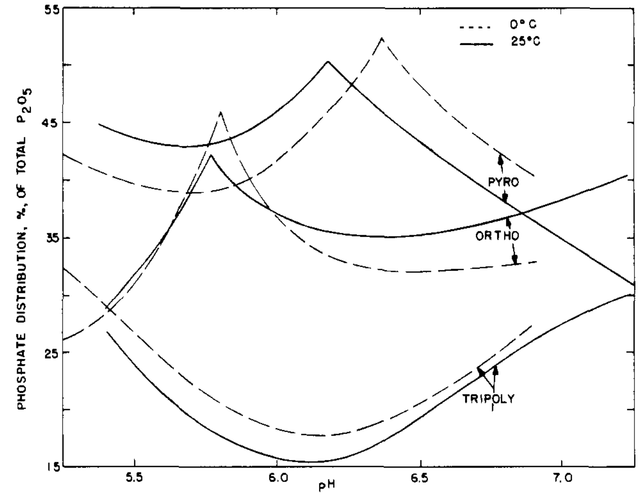

Figure 6. Distribution of phosphate species in the system $CO(NH_2)_2$–NH_3–H_3PO_4–$H_4P_2O_7$–$H_5P_3O_{10}$–H_2O at 0 and 25 °C.

both temperatures. The pH of the saturated solutions is related to the ratio NH_4-N:P_2O_5 (R) at 0 °C by

$$\mathrm{pH} = (-1.27 \pm 0.14) + (24.6 \pm 0.5)R \quad (3)$$

with a correlation coefficient of 0.997 and a prediction standard deviation of 0.04 pH unit, and at 25 °C by

$$\mathrm{pH} = (-1.76 \pm 0.17) + (26.7 \pm 0.6)R \quad (4)$$

with a correlation coefficient of 0.996 and a prediction standard deviation of 0.05 pH unit.

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Densities, Viscosities, and Conductances of Saturated Solutions in the Systems Mercuric Chloride–Hydrogen Chloride–Water, Mercuric Chloride–Potassium Chloride–Water, and Mercuric Chloride–Indium Chloride–Water at 25.0 °C

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The densities, viscosities, and specific conductances of saturated solutions in the systems mercuric chloride–hydrogen chloride–water, mercuric chloride–potassium chloride–water, mercuric chloride–indium chloride–water were determined at 25.0 °C. The same properties were determined for aqueous solutions of indium trichloride in the concentration range 0.642–5.157 N and for mercuric chloride solutions from 0.001 N to saturation, both at 25.00 °C.

Introduction

In a previous paper (*1*), the phase diagram of the system: $HgCl_2$-$InCl_3$-H_2O was determined. The solubility curve of $HgCl_2$(s) indicates a large increase in solubility, from 6.7% to 33%, by the addition of a relatively small amount of indium chloride. This phenomenon is even more marked in the system $HgCl_2$-HCl-H_2O (*2*), where addition of ~8% HCl produces a tenfold increase in solubility. In both systems, no compound separates in the solid state, the solid phase being $HgCl_2$ anhydrous. This is in contrast to the system $HgCl_2$-KCl-H_2O where three double salts have been reported by Linke and Seidell (*3*). Here the solubility increases to 25% with the addition of 5% KCl. It may be expected that this increase in solubility of mercuric chloride would be the result of a change of the molecular species present in solution, in which case measurements of conductance and viscosity of solutions saturated with mercuric chloride (or double salt) would be of value.

Table I. Specific Conductances (κ), Densities, and Viscosities (η) of Solutions of Hydrogen Chloride Saturated with $HgCl_2$(s) at 25.0 °C

$[HgCl_2]$, wt %	[HCl] wt %	[HCl] M	density, g mL^{-1}	η^a	κ, Ω^{-1} cm^{-1}
16.4	1.53	0.487	1.160	1.121	0.1370
27.3	2.90	0.1036	1.303	1.229	0.2951
34.0	3.74	1.449	1.413	1.355	0.3817
41.0	4.56	1.926	1.540	1.541	0.4543
57.7	6.55	3.464	1.929	2.530	0.5102
65.4	8.05	5.145	2.331	5.059	0.4100
69.3	9.64	6.499	2.459	6.728	0.3620
69.2	11.0	8.373	2.776	9.441	0.3170

[a] $\eta_{H_2O} = 1.000$.

Table II. Specific Conductances (κ), Densities, and Viscosities (η) of Solutions of Indium Trichloride Saturated with $HgCl_2$(s) at 25.0 °C

$[InCl_3]$ wt %	$[InCl_3]$ M	$[HgCl_2]$, wt %	density, g mL^{-1}	η^a	κ, Ω^{-1} cm^{-1}
2.81	0.148	15.9	1.166	1.213	0.019 95
8.91	0.518	20.3	1.284	1.458	0.034 47
9.25	0.584	25.0	1.397	1.796	0.043 0
13.5	0.903	27.1	1.479	2.108	0.045 3
16.8	1.191	28.8	1.568	2.568	0.044 71
20.6	1.553	29.7	1.667	3.347	0.039 94

[a] $\eta_{H_2O} = 1.000$.

Experimental Section

In each of the three systems, several saturated solutions were prepared at 25.0 °C. The phases were separated by centrifuge at room temperature because of the high viscosity of the solutions. This introduces a small uncertainty in the temperature and therefore in the composition of the saturated solutions but would not change the nature of the solid phase(s). The liquid phases were analyzed for Hg^{2+} as HgS and then for Cl^- as AgCl. Solutions containing indium were made strongly acidic with sulfuric acid, so that indium sulfide would not separate out on addition of hydrogen sulfide. The second cation in each system was obtained by difference. The densities, viscosities, and specific conductances of the saturated solutions were measured at 25.0 °C. These data are given in Tables I–III, where the weight percentages of hydrogen chloride, potassium chloride, and indium chloride are also expressed as molarities in order to compare the properties of the saturated solutions with those of aqueous solutions of hydrogen chloride, potassium chloride, and indium chloride of the same molarities. These latter values were taken from the literature in the case of hydrogen chloride (*5*) and of potassium chloride (*6*), while

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