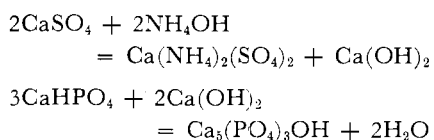


**Table IV. Calculated Compositions of Ammoniated Superphosphates**

No.	Composition, Millimoles/10 Grams of Fertilizer								
	$\text{NH}_4\text{NO}_3$	$\text{NH}_4\text{H}_2\text{PO}_4$	$(\text{NH}_4)_2\text{SO}_4$	$\text{CaSO}_4$	$\text{Ca}(\text{NH}_4)_2(\text{SO}_4)_2$	$\text{H}_2\text{O}$	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$	$\text{CaHPO}_4$	$\text{Ca}_3(\text{PO}_4)_2\text{OH}$
247	25	3.5	0	26	0	0	6.5	3.5	1.0
248	25	9	0	19	4	0	7.5	2.1	
249	14	5	0	2	15	0	6.6	4.5	
250	10	2	10	0	10	0	5.8	5.8	
251	54	0	0	1	0	16.4	1.0	0.0	
253	39	19	1	0	0	0	20 <sup>a</sup>	0.6 <sup>a</sup>	

<sup>a</sup> Without allowance for  $\text{Ca}(\text{NH}_4)_2(\text{HPO}_4)_2 \cdot \text{H}_2\text{O}$ .

The conversion of anhydrite to syngenite and ammonium sulfate during the ammoniation results in the formation of apatite. Although the reactions are complex, they may be represented as



The solubility product of calcium sulfate,  $a_{\text{Ca}} \cdot a_{\text{SO}_4}$ , shows that the decomposition of gypsum or anhydrite is independent of pH. The solubility product of apatite,  $a_{\text{Ca}}^5 \cdot a_{\text{H}_2\text{PO}_4}^3 / a_{\text{H}^+}^7$ , indicates that the limiting calcium ion concentration above which it will form decreases rapidly with rising pH. Thus, although calcium sulfate and monoammonium phosphate may be a stable salt pair at low degrees of

ammoniation, there is a critical level of ammoniation above which calcium sulfate acts as a continuous source of calcium for the formation of the more basic phosphate.

The effect of ammoniation on the composition of the water-insoluble phosphates in ammoniated ordinary superphosphate is shown in Figure 3. There is good correlation between the amount of phosphate calculated to be present as apatite and the fraction that is insoluble in alkaline ammonium citrate solution, and either quantity can be used as an index of the degree of ammoniation of ammoniated ordinary superphosphate. The fraction of phosphate soluble in neutral ammonium citrate is much less significant. The discrepancies between solubilities in neutral and alkaline citrate solutions indicate that 60 to 70% of the

apatitic material is soluble in the neutral reagent.

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**FERTILIZER NUTRIENT RELEASE**

**Composition and Dissolution of Ammoniated Superphosphate Fertilizers Containing Potash**

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Potassium chloride added to superphosphate has little effect on the form of the phosphate when the mixture is ammoniated, but reacts with ammonium nitrate to form potassium nitrate and ammonium chloride. Ammoniation of mixtures of ordinary superphosphate and potassium chloride forms potassium syngenite,  $\text{CaK}_2(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$ , and at high degrees of ammoniation as much as half the potassium is converted to this water-insoluble salt. When diammonium phosphate is the ammoniating agent, insoluble sulfates such as syngenite are dissolved. Ammoniation of concentrated superphosphates containing added potassium chloride yields monopotassium phosphate. Dissolution of potassium-containing ammoniated superphosphates releases solutions of ammonium and potassium phosphates with pH's in the range 2.5 to 4.5; the pH rises as the degree of ammoniation of the superphosphate is increased.

THE composition and dissolution of a series of ammoniated superphosphate fertilizers containing no potassium salts have been described (2). Similar studies were made of fertilizers prepared by ammoniating mixtures of superphosphate and potassium chloride.

The compositions of the fertilizers are shown in Table I. Fertilizers 302, 303,

199, and 304 were experimental TVA materials, field-tested as sources of phosphorus by Wright, Lancaster, and Anthony (3). Fertilizers F15 and T6 were domestic commercial products, and EA and EB were European commercial products.

The experimental procedure was the same as in the previous study (2). The

results of the fractional 1-hour elution experiments are presented in Figure 1; no fractional elutions were made of the European fertilizers. The amounts of the component elements removed in a single 24-hour elution are shown in Table II.

Comparison of the data in Figure 1 with those for comparable potassium-

**Table I. Composition of Fertilizers**

No.	Grade	Composition, %							Fraction, %, of P Soluble in		
		N		P	S	Ca	K	Cl	Ammonium Citrate		
		Total	NH <sub>3</sub>						H <sub>2</sub> O	Neutral	Alkaline
302	6-5.2-10 (6-12-12)	5.8	3.6	5.9	7.9	14.0	10.3	9.8	62	94	93
303	6-5.2-10 (6-12-12)	6.3	4.3	5.9	7.9	13.9	10.2	9.4	43	93	72
199	6-5.2-10 (6-12-12)	6.5	5.1	6.2	7.7	13.6	10.3	9.5	27	96	51
304	6-5.2-10 (6-12-12)	5.6	4.9	5.7	7.8	13.7	10.3	9.1	11	91	32
EA	10-4.4-8.3 (10-10-10)	9.4	8.6	4.8	12.4	9.6	9.5	8.8	53	98	96
EB	7-6.1-11.5 (7-14-14)	6.8	6.4	6.1	10.2	11.4	11.3	10.4	46	76	68
T6	6-10.5-20 (6-24-24)	4.6	3.6	8.5	3.4	6.9	20.7	19.6	76	98	...
F15	15-6.6-12.5 (15-15-15)	16.0	12.4	6.5	6.0	3.1	12.5	11.3	76	98	95

free fertilizers (2) reveals a broad similarity in the behavior of the two sets of fertilizers. The rapid initial release of ammonium and nitrate was a prominent feature; in this series it was accompanied by a similar release of chloride. The concentrations of chloride in the eluates were about equal to those of ammonium, indicating dissolution of ammonium chloride. Potassium concentrations were high in the early stages of the dissolution, but, although all four fertilizers contained the same amount of potassium, there was a marked tendency for both the maximum concentration and total amount of potassium in the eluate to decrease as the ratio of ammonium to potassium in the original fertilizer increased with increasing ammoniation. In fertilizer 304, 48% of the potassium was retained in the fertilizer after 24 hours' elution (Table II). In fertilizer 302, the potassium concentration passed through a maximum, indicating that there was some precipitation of potassium during the dissolution. This maximum is not necessarily connected with that in the phosphate curve, which may be due to the initial suppression of the dissolution of monoammonium phosphate by the high ammonium concentration, as was observed previously (2).

The relationship between the fraction of phosphate soluble in water and the degree of ammoniation of the four TVA fertilizers is plotted in Figure 2 with the same data for the potassium-free fertilizers. The two sets of points lie close to a common line, indicating that the effect of incorporation of potassium is slight. At higher degrees of ammoniation the potassium may cause some decrease in the water solubility, but the degree of ammoniation is the controlling factor.

The amounts of the individual salts in the original fertilizers cannot be calculated from the data in Table II by the

**Table II. Compositions of Original Fertilizers and Amounts of Components Removed by Leaching**

No.		Millimoles/10 Grams of Fertilizer							Degree of Ammoniation <sup>a</sup>
		NH <sub>4</sub>	NO <sub>3</sub>	K	Cl	P	Ca	S	
302	Total	25.7	15.7	26.4	27.6	19.2	34.9	24.6	0.52
	Removed	25.7	15.7	24.6	26.8	9.6	2.4	1.2	
303	Total	30.7	14.2	26.1	26.5	19.1	34.6	24.6	0.87
	Removed	30.0	13.6	19.7	26.2	4.7	1.2	3.4	
199	Total	36.4	10.0	26.4	26.8	19.9	34.1	24.0	1.33
	Removed	33.6	9.3	14.6	26.2	3.6	0.9	4.7	
304	Total	35.0	5.0	26.4	25.7	18.5	34.2	24.3	1.62
	Removed	31.4	5.0	13.8	25.7	0.7	0.4	7.2	
F15	Total	88.5	25.8	32.0	31.9	21.0	7.8	18.7	...
	Removed	87.8	25.0	31.2	31.6	14.9	0.2	18.2	
T6	Total	26.0	7.1	53.0	55.3	27.5	17.2	10.6	...
	Removed	25.6	6.4	51.5	53.9	19.0	2.9	1.9	
EA	Total	61.4	4.3	24.3	24.8	15.5	24.0	38.7	...
	Removed	61.4	4.1	12.9	24.7	8.2	nil	18.0	
EB	Total	45.7	1.4	28.9	29.3	19.7	28.5	31.8	...
	Removed	44.0	1.4	13.8	29.3	9.0	nil	9.4	

<sup>a</sup> Mole ratio (NH<sub>4</sub> - NO<sub>3</sub>):P.

methods described previously (2) because of the greater complexity of the mixtures. Wide variations are possible in the proportions of ammonium and potassium in mixed crystals of (NH<sub>4</sub>,K)Cl, (NH<sub>4</sub>,K)<sub>2</sub>SO<sub>4</sub>, and syngenite, Ca(NH<sub>4</sub>,K)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O, and similar substitution of potassium for ammonium may take place to some extent in monoammonium phosphate. The changes in the x-ray diffraction spacings caused by these substitutions are too small to permit reliable estimation of the composition of the materials. Estimation of the separate amounts of ammonium and potassium associated with each anion thus is impracticable, and the composition of the fertilizers can be stated only in general terms.

The results in Table III are based upon several assumptions. The total ammonium and potassium are ap-

portioned among the chloride, nitrate, sulfate, and water-soluble phosphate. The sulfate is apportioned between calcium sulfate and syngenite, Ca(NH<sub>4</sub>,K)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O, since the relative amounts of ammonium and potassium in the syngenite and in the free ammonium and potassium sulfates in fertilizer 304 are not known. The data in Table III cannot be converted to a weight percentage basis because the formulas, and hence the formula weights, are not known. The compositions of the water-insoluble phosphates are based on the assumption that the calcium content of the fertilizer can be apportioned among dicalcium phosphate, apatite, and the sulfates (2). The data in the fertilizer series 302 through 304 show that the amount of syngenite increases with increased ammoniation until the free calcium sulfate is depleted, as in the previous study (2).

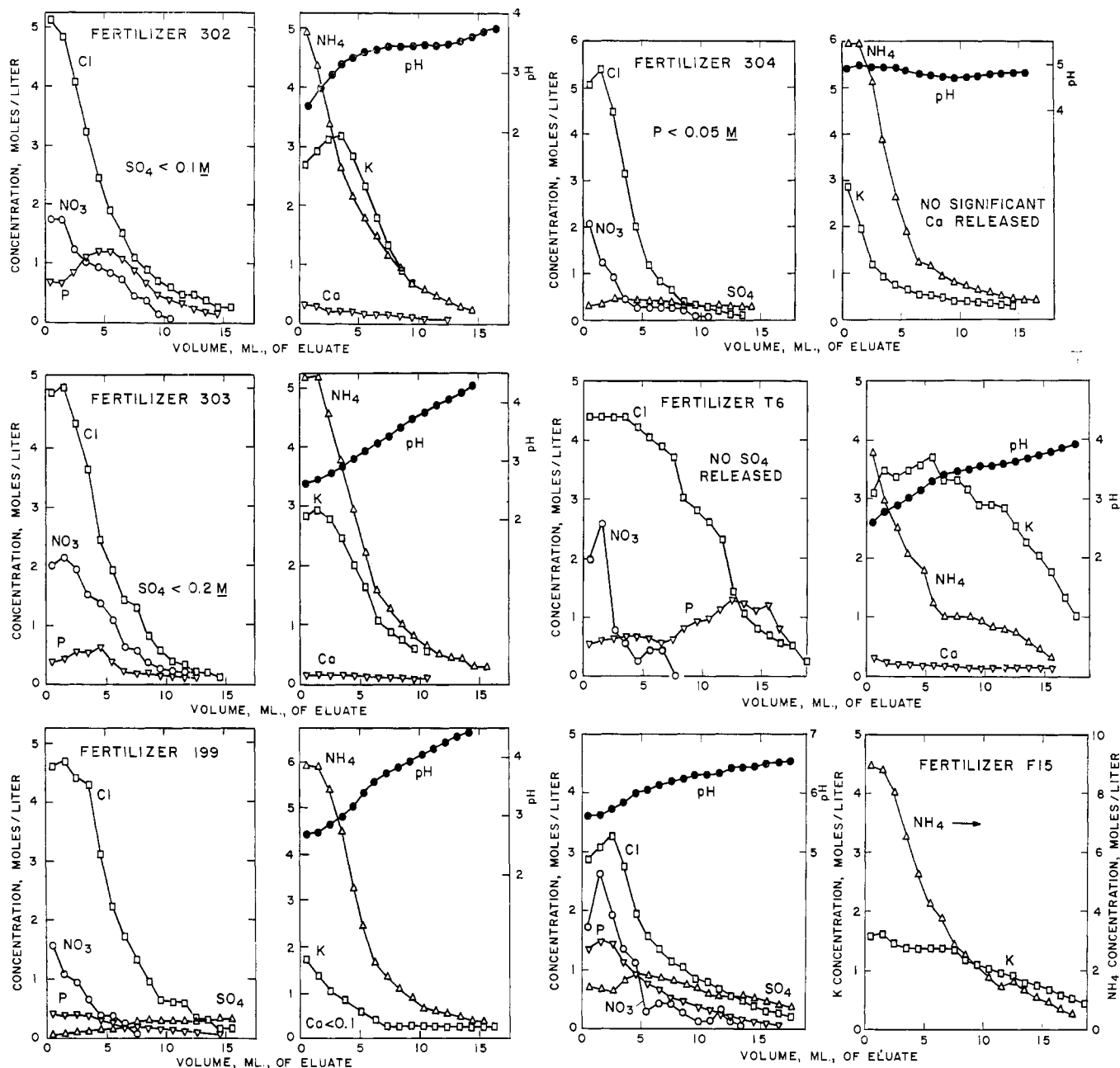


Figure 1. Dissolution of fertilizers

Ten-gram samples leached with 1 ml. of water per hour; receivers changed every hour

X-ray examination of the residues showed that the potassium in the residues was present as potassium syngenite which did not hydrolyze to gypsum during the dissolution. The increase in the amount of insoluble potassium with increasing ammoniation indicates that ammoniation formed the syngenite in the original fertilizer, releasing calcium which reacted to form basic calcium phosphates.

The estimated amounts of gypsum and syngenite in the residues are included in Table III. There was more syngenite in the residue of fertilizer 302 than in the original fertilizer, and the low potassium concentrations in the early stages of dissolution (Figure 1) probably are due to the formation of syngenite during the

dissolution. Calcium sulfate is consumed in the formation of syngenite. The free calcium sulfate in fertilizer 303 remained constant during the dissolution, and that in fertilizers 199 and 304 increased, showing that some syngenite was dissolved. Considerable gypsum was formed in the dissolution of fertilizers EA and EB, in which a considerable fraction of the syngenite probably was in the ammonium form. The formulation of these European fertilizers is not known, but the data in Table II suggest that it included some ammonium sulfate which formed syngenite with the calcium sulfate. During the dissolution of these fertilizers the granules collapsed into a wet paste which later solidified to a plasterlike mass and then a hard and

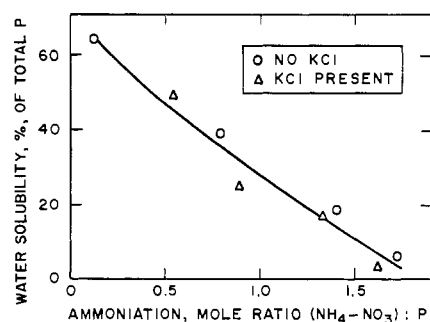


Figure 2. Effect of ammoniation on water solubility of phosphorus in superphosphate

resistant residue of gypsum, a behavior that was not observed with the other fertilizers.

The formulations of fertilizers F15 and T6 are given in Table IV. The results of a quantitative x-ray analysis of these materials, as described by Ando *et al.* (1), are presented in Table V for comparison with the data in Table II. Fertilizer T6 can be regarded as an ammoniated concentrated superphosphate to which diammonium phosphate and potassium chloride have been added, with ammoniacal ammonium nitrate solution as the principal ammoniating agent. Most of the potassium is present as the chloride and monopotassium phosphate. The influence of these compounds is evident in the dissolution curves in Figure 1, where potassium and chloride are the dominant ions. The release of phosphate is suppressed in the early stages of dissolution where the potassium and ammonium concentrations are highest, delaying the dissolution of monopotassium and monoammonium phosphates. The high initial ammonium concentration probably reflects the removal of ammonium chloride; the free ammonium chloride cannot be distinguished from that found by x-ray to be present in solid solution with potassium chloride.

The amounts of sulfates appear to be overestimated in the x-ray analysis, but it is apparent that a considerable fraction of the sulfate was present as free ammonium and potassium sulfates and calcium sulfate. Relatively little sulfate was released during the dissolution, but almost all the potassium and ammonium were removed. The absence of syngenites contrasts with the previous observations which show that free ammonium and potassium sulfates are not found together with free calcium sulfate in ammoniated ordinary superphosphates. The reason for the existence of ammonium and potassium sulfates in fertilizer T6 is not fully understood. Table IV shows that sulfate was introduced as sulfuric acid and not as calcium sulfate, and the calcium sulfate identified by x-ray must therefore have been precipitated during the manufacture of the fertilizer, thus providing less favorable conditions for formation of syngenite than when it is added as a component of ordinary superphosphate. The composition of fertilizer T6 shows that in the presence of potassium chloride the ammoniation of concentrated superphosphate forms monopotassium phosphate, which is not found in fertilizers prepared from ordinary superphosphate.

The absence of syngenite and the small amount of calcium sulfate in fertilizer F15 are also significant, since this fertilizer was formulated with ordinary superphosphate. The absence of syngenite appears to result from the presence of diammonium phosphate,

**Table III. Calculated Compositions of Fertilizers**

Component	Millimoles/10 Grams of Initial Fertilizer					
	302	303	199	304	EA	EB
In initial fertilizer						
NH <sub>4</sub> H <sub>2</sub> PO <sub>4</sub> <sup>a</sup>	9.6	4.7	3.6	0.7	8.2	9.0
KNO <sub>3</sub> <sup>a</sup>	15.7	14.2	10.0	5.0	4.3	1.4
(NH <sub>4</sub> ,K)Cl	27.6	26.5	26.8	25.7	24.8	29.3
Ca(NH <sub>4</sub> ,K) <sub>2</sub> (SO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	0.4	5.7	11.2	9.3	14.5	14.3
CaSO <sub>4</sub>	23.8	13.2	1.6	0.0	0.0	0.0
(NH <sub>4</sub> ,K) <sub>2</sub> SO <sub>4</sub>	0.0	0.0	0.0	5.7	19.4	6.3
P						
As CaHPO <sub>4</sub>	8.0	12.5	8.6	7.2	4.0	5.4
As Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>3</sub> OH	1.6	1.9	7.7	10.6	3.3	5.3
In residue						
Ca(NH <sub>4</sub> ,K) <sub>2</sub> (SO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	0.9	4.0	7.3	8.1	5.7	7.6
CaSO <sub>4</sub>	21.6	13.3	4.7	0.9	9.3	7.3
Distribution, % of P						
Water-soluble	50	25	18	4	53	46
CaHPO <sub>4</sub>	42	65	43	39	26	27
Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>3</sub> OH	8	10	39	57	21	27

<sup>a</sup> All water-soluble phosphate assumed to be NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> and all nitrate assumed to be KNO<sub>3</sub>.

which dissolves to give solutions of pH 5.5 to 6.5. Under these conditions the calcium activity in equilibrium with dicalcium phosphate or apatite will be very much lower than that in equilibrium with calcium sulfate, so that the calcium sulfate dissolves. The nearly complete removal of sulfate during the leaching indicates that all the calcium sulfate was dissolved in this way, probably causing precipitation of water-soluble phosphate.

Ammonium and potassium were the only two cations dissolved from fertilizer F15. The high ammonium concentration in the early stages of leaching probably represented the dissolution of the double salt 2NH<sub>4</sub>NO<sub>3</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> along with the ammonium chloride. Except for the rise in concentration of sulfate, which probably represented a delay in dissolution of free ammonium sulfate by a common-ion effect, the concentrations of all the ions fell steadily as the dissolution progressed. The insoluble residue of this fertilizer was composed of basic calcium phosphates.

### Conclusions

With the exception of potassium syngenite, the various double salts and mixed crystals that have been identified in ammoniated superphosphate fertilizers by x-ray methods appear to have little effect on the behavior of the fertilizers. Soluble salts such as chlorides and nitrates are rapidly removed from the fertilizer as ammonium and potassium salts in the early stages of dissolution. The dissolution of soluble phosphate present as monoammonium or monopotassium phosphate is initially suppressed by the common-ion effect, and most of the phosphate is released in the later stages of dissolution.

As phosphate fertilizers, these materials may be regarded as sources of concentrated ammonium and potassium

**Table IV. Fertilizer Formulations**

Component	Fertilizer	
	F15	T6
	Grade	
	15-	
	6.6-12.5	6-10.5-20
	(15-15-15)	(6-2-4)
Component	Lb. Component/Ton Fertilizer	
Nitrogen solution (44% N)	534	190
(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	404	200
Ordinary superphosphate	222	0
Concentrated superphosphate	80	800
KCl	496	820
H <sub>2</sub> SO <sub>4</sub> (66° Be.)	329	25

**Table V. Fertilizer Compositions Determined by X-Ray Analysis**

	Millimoles/10 Grams Fertilizer	
	F15	T6
NH <sub>4</sub> Cl	20.5	13.1
NH <sub>4</sub> NO <sub>3</sub>	16.5 <sup>a</sup>	0.0
NH <sub>4</sub> H <sub>2</sub> PO <sub>4</sub>	8.7	7.0
(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	9.8	0.0
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	10.4 <sup>a</sup>	1.5
KCl	24.0	37.5
KNO <sub>3</sub>	1.3	0.0
KH <sub>2</sub> PO <sub>4</sub>	0.6	2.3
K <sub>2</sub> SO <sub>4</sub>	1.5	8.8
Ca(NH <sub>4</sub> ) <sub>2</sub> (SO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	0.0	0.3
CaK <sub>2</sub> (SO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	0.0	0.6
CaSO <sub>4</sub>	2.2	5.2
Ca(H <sub>2</sub> PO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	0.0	2.0
CaHPO <sub>4</sub>	2.2	4.4
Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>3</sub> OH	1.0	0.6

<sup>a</sup> Includes that present as 2NH<sub>4</sub>NO<sub>3</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.

phosphate solutions with pH's between 3 and 6, depending on the degree of ammoniation; the pH is above 4.5 only when diammonium phosphate is present in the fertilizer. Very lightly ammoniated materials that still contain

monocalcium phosphate may initially release solutions with pH's below 2 that contain appreciable amounts of calcium.

Potassium syngenite, which is relatively insoluble in water, might be expected to act as a slowly soluble source of potash. Results of exploratory

greenhouse tests, however, show little indication of this effect.

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## FERTILIZER TECHNOLOGY

# Heat of Ammoniation of Superphosphoric Acid

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In the temperature range 100° to 200° C. the amount of ammonia absorbed by superphosphoric acid (33% P, 76% P<sub>2</sub>O<sub>5</sub>) rapidly enough to give a measurable heat effect increased with rising temperature. The heat of ammoniation of superphosphoric acid was measured at base temperatures of 100°, 125°, and 180° C.; the respective maximum ammonia concentrations were 1.24, 3.54, and 4.37 pounds of NH<sub>3</sub> per 20 pounds of P<sub>2</sub>O<sub>5</sub>, and the integral heats of ammoniation at the respective maximum ammonia concentrations were 3548, 3023, and 2862 B.t.u. per pound of NH<sub>3</sub>. Ammoniation made only slight changes in the distribution of the phosphate species.

AMMONIUM polyphosphate, a mixture of the ammonium salts of ortho- and condensed phosphoric acids, is a new and promising fertilizer material (2, 7), prepared by ammoniation in a closed vessel at about 200° C. of superphosphoric acid that contains about 33% P (76% P<sub>2</sub>O<sub>5</sub>). Knowledge of the heat of ammoniation of superphosphoric acid is useful in plant design calculations.

#### Apparatus

The calorimeter was built around a stainless steel 300-ml. Parr oxygen bomb, fitted with a single gas port and a stainless steel stirrer driven at 590 r.p.m. A 100-ohm electrical calibrating heater was wound in a spiral groove in the outer surface of the bomb. The bomb was immersed in a bath of molten wax for measurements at 100° and 125° C. and of silicone oil for measurements at 180° C., the highest practicable base temperature for the assembly.

The liquid bath was contained in a stainless steel vacuum flask and was stirred by a stream of nitrogen from a sparger ring. The flask was fitted with a Transite lid that supported three stainless steel radiation shields. An electrical heater was wound on the outer surface of the vacuum flask, and the assembly was placed in an electrically heated oven that was maintained at a temperature suitably lower than that at which a run was made. The vacuum flask was pumped continuously.

The ammonia was admitted as a gas from a 300-cc. liquid ammonia tank through a 1/8-inch stainless steel tempering coil that passed through the oven and was attached to the top of the bomb. The pressure in the system was measured at the valve on the ammonia tank.

A precision temperature controller actuated by a resistance thermometer maintained the outer surface of the vacuum flask at the base temperature of each run. Each run was started with the calorimeter (bomb and liquid bath) within 0.1° C. of the base temperature. Calorimeter temperatures during a run were measured at 1-minute intervals with a calibrated platinum resistance thermometer in a glass case in the liquid bath. Straight-line cooling curves had slopes of 0.006° per minute at 180° C. and 0.054° per minute at 197° C.; the calorimeter thus was suitable for obtaining data of the reliability required for design calculations.

#### Reagents

The liquid ammonia from a synthesis plant was used as received.

Three samples of superphosphoric acid were used. The acid used for runs at 100° C. was not heat-treated sufficiently

to equilibrate the phosphate species (4, 5).

The acid for runs at 125° C. was a product of a demonstration-scale superphosphoric acid plant, and that for runs at 180° C. was made by adding reagent grade phosphoric oxide to reagent grade orthophosphoric acid. Each acid was heated in a sealed borosilicate glass container to 350° C. in 20 minutes and held at 350° C. another 10 minutes to equilibrate the phosphate species. Both acids then had the same P<sub>2</sub>O<sub>5</sub> content and the equilibrium distribution of species within the limits of error of chromatographic analysis. The compositions of the acids are listed in Table I.

#### Procedure

The bomb was charged with a weighed amount of superphosphoric acid and heated to the base temperature. The calorimeter then was calibrated electrically, ammonia was added to give a

Table I. Initial and Ammoniated Superphosphoric Acids

Sample	Composition, %				Distribution, % of P <sup>a</sup>					
	N	P	NH <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	Ortho	Pyro	Tri	Tetra	Penta	Other
A										
Initial	0.0	33.23	0.0	76.15	61.8	...	...	...	...	...
Final	3.7	31.74	4.5	72.73	...	...	...	...	...	...
B										
Initial	0.0	33.12	0.0	75.88	50.8	40.2	7.3	1.2	...	0.5
Final	9.8	29.17	11.9	66.85	52.4	32.2	10.6	3.2	...	0.6
C										
Initial	0.0	33.12	0.0	75.88	49.1	39.3	8.6	2.2	...	0.8
Final	11.4	28.14	13.9	64.49	50.7	34.6	9.7	3.4	0.9	0.5
Equilibrium (5, 6)	0.0	33.17	0.0	76.00	49.0	42.8	7.1	1.0	0.1	0.02

<sup>a</sup> After ortho- and pyrophosphates, prefixes denote *n* in formula H<sub>n+2</sub>P<sub>n</sub>O<sub>3n+1</sub>; "other" denotes species too large to move on chromatographic paper.