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FEED TECHNOLOGY

Handling Phosphoric Acid in Formula Feeds

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Phosphoric acid has proved acceptable as a nutrient in animal rations, but its handling in milling equipment has been questioned, because of its corrosive nature. A technique was developed for distributing acid uniformly throughout a commercial feed. Corrosion was observed as weight losses in mild steel strips secured to mixing, conveying, and pelleting equipment. Initially those areas in the mixer subjected most to abrasion were also affected most by corrosion from acid-containing feeds; however, areas of little friction eventually also showed considerable corrosion. With low-level (1.25%) acid addition, test strips showed weight changes normally suitable for materials used in feed milling equipment, except in the pellet mill conditioner chamber. When mixing high levels of acid (up to 35%) with limestone and soybean oil meal to form a dry premix, it was evident that the mixer should be made of resistant material. Once mixed, high-level acid carriers were used as an ingredient in feeds with no change in processing from the basal ration.

OF THE 37,000,000 TONS of formula feeds manufactured annually in this country, it is estimated (7) that about 1.8% are inorganic phosphorus additives. This is equivalent to 173,000,000 pounds of the element phosphorus. Defluorinated domestic rock phosphates, bone meal, colloidal phosphates, imported low-fluorine rock phosphates, and other phosphorus-containing materials are used to furnish the essential nutrient.

Phosphate supplements are commonly supplied as a dry material, in 50- or 100-pound bags with attendant problems of container disposal and material loss from breakage and incomplete emptying. Bulk handling is successfully used by some feed manufacturers and may be more generally accepted if attention is given to producing a granular product containing little dust. Liquid phosphorus supplements, as are used by the soft drink industry, have been proposed for formula feeds. The economic aspects both as to unit phosphate cost and handling cost have been covered by Maddy (3). The questions of nutritive value and availability have been answered for poultry by Titus (4) and for livestock by Menzies (5), and the effect on nutrients under adverse storage conditions by the Wisconsin Alumni Association, as reported by Mehring (4). The mechanics of handling liquid phosphoric acid have been studied in the

pilot formula feed plant at the Kansas Agricultural Experiment Station.

Liquid additives supplementing dry feeds and even liquid feeds are not new to the industry (2). Cane molasses with a pH of 5.0 containing lactic acid, fish solubles with a pH of 4.5 containing sulfuric acid, and inedible animal and vegetable fats containing fatty acids, are in contact with a great deal of mixing and conveying equipment in certain feed mills. It has not been found necessary to replace equipment or specify stainless steel when using liquids containing weak organic acids or low concentrations of mineral acids buffered by proteins or their hydrolyzates.

Phosphoric acid is corrosive to mild steel, copper, brass, and bronze and is sufficiently hazardous to require precaution in handling. The common commercial products contain the equivalent of 75 to 85% orthophosphoric acid. Material used in this study contained 23.8% phosphorus and had a density of 13.3 pounds per gallon. Under the experimental conditions reported, about 1400 pounds of acid were added to feeds at an average level of 1.25%, with 90% of the feed being prepared in pelleted and crumbled form.

Equipment

Stainless steel holding and dispensing equipment was installed to supply acid

to a batch horizontal mixer. Figure 1 shows a schematic drawing of the reservoir with connections to the mixer. Air pressure, superimposed on the liquid in the acid feed tank, was necessary to maintain a spraying action during the acid addition. An air bypass line, below the tank, permitted pressure to spray nozzles when liquid was not being dispensed. A header, to which were attached four spray nozzles, carried acid into the mixer either at position A or B as seen in Figure 2. Position A was above the agitators on the up side of the feed movement and in effect, feed was thrown against the header in this position. At B, directly across the mixer from A, the header was about 10 inches above the feed flow during mixing, because gravity and centrifugal movement of the spirals folded material into the mixer trough below this position.

Phosphoric acid was brought to the plant warehouse in lacquer-lined 55-gallon steel drums and transferred by air pressure and plastic tubing to 5-gallon carboys to be conveyed into the mill. Coveralls and goggles were used at all times. A shower stall was within 40 feet of the liquid acid for emergency use. The mixer was 15 cubic feet in capacity, 5 feet long × 24 inches wide with a double ribbon agitator, a 10-inch outlet, and operating at 69 r.p.m. with a 5-hp. motor. After discharge by the air

cylinder-operated gate, mixed feed was spouted to an elevator boot and taken 49 feet to a sifter and from this by jack-leg elevator and screw conveyor to a bin over the pellet mill.

The question of dimensional change in the milling equipment was studied by securing coded mild steel strips to the mixer sides, outer and inner spirals (Figure 3), elevator cups, spouting and pellet mill conditioner chamber, side plate, and paddles. The test strips measured $\frac{1}{2} \times 3 \times \frac{1}{16}$ inches and less the holes used for bolting to equipment, the surface area exposed to abrasion or corrosion was 1.8825 square inches. Plastic insulation strips and washers were used to prevent contact of the strips with the metal equipment.

Experimental Procedure and Results

Distribution of Acid in Feed. The basal ration used was formulated to be fed with equal parts of grains, to provide a balance of known nutrients, for egg production. Supplemental calcium was to be available in the laying house as grit. Acid was calculated into the formula on a phosphorus equivalent basis replacing a bone meal product containing 14% phosphorus specified in the basal ration. When acid was substituted for bone meal, the calcium accompanying phosphorus in bone meal was supplied as limestone, supplementing that already in the formula. The basal ration and changes obtained with the substitution of acid for bone meal are shown in Table I.

When the acid feed tank was charged with the calculated amount of acid for a mix, it was put under air pressure and released into the bulk of the feed during the action of mixing and immediately after discharge of the material from the scale hopper. A drop of pressure on the head pressure gage indicated that all the acid had been ejected. The vitamins and small ingredients not binned above the scale hopper were then added, and a mixing time of 6 minutes followed.

The first problem, and one requiring continuous attention, was to obtain a uniform blending of liquid and dry feed. It was soon apparent that with the dispensing header located in position A, no adjustment of nozzle position or air pressure could prevent an increase in the amount of material retained on a Tyler No. 9 screen. In this position, excessive clogging of spray nozzles was also observed. Apparently the throwing action of the agitators carried material over the header and the nozzles, preventing a spraying action. After acid was dispensed, the wetted nozzles became encrusted with dust and feed material, resisting air pressure to free them.

The acid header at position B was therefore studied. Variations in the direction of nozzles and amount of air pressure were analyzed by measuring the

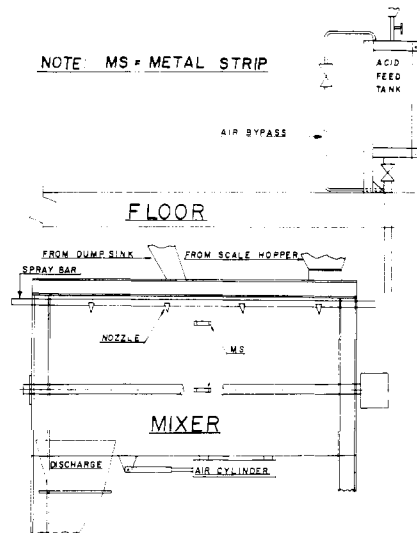


Figure 1. Schematic diagram of reservoir with connections to the mixer

SPECIFIC DYNAMIC FLOW LINE

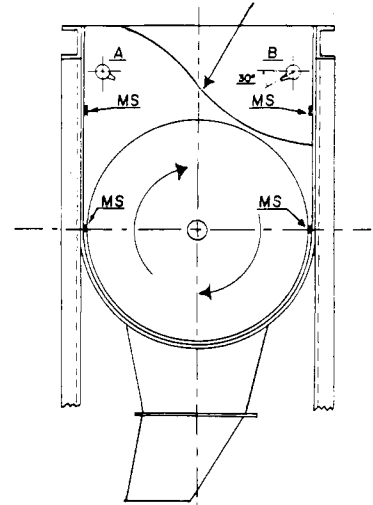
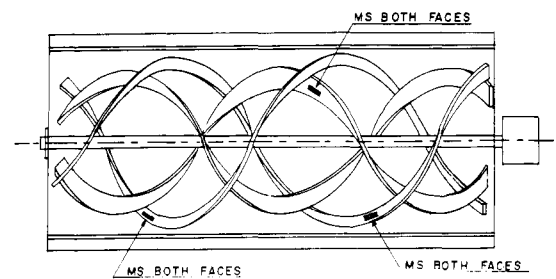


Figure 2. Header with positions A and B

Figure 3. Outer and inner spirals with steel strips attached



uniformity of particles retained on Tyler screens as compared with those of the basal ration. At all nozzle directions and air pressures, some balling of acid and feed occurred when flat, wide angle, low impact velocity sprays were used. Nozzles did not clog, however, in this position. Full jet cone-type sprays gave no improvement. Flat, atomizing nozzles of the type TT-8002 (Spraying Systems Co.) made from stainless steel with 50-mesh Monel strainer were tried and improvement was noted at once. After it was determined that least balling occurred when the nozzle angle from header to horizontal was 30° , all subsequent work was performed with these conditions of optimum uniformity in the acid distribution.

In position B, with spray nozzles directed at 4 o'clock and acid encountering rapidly moving feed, little change in the sieve size of the mixed material was noted (see Table II). High concentrations of acid and fine feed material were observed in the overs of the No. 24 sieve, which are similar in size to the bulk of soybean oil meal used. These were grayish, flattened particles, which were strongly acid to taste. The pH of these particles was 2.0 to 2.2. Sieving was not performed at once after mixing, but samples were allowed to remain overnight in uncovered cans, and after some drying and hardening of acid feed balls, 200 grams were shaken for 1 minute on a Tyler Rotap. These observations showed that acid agglomeration with feed by adhesion was confined

Table I. Laying Mash Concentrate with and without Phosphoric Acid

Ingredient	Basal Ration	With Acid
Yellow corn meal	400	400
Ground grain sorghums	300	300
Wheat standard middlings	450	450
Ground oats	50	50
Soybean oil meal	400	400
Dehydrated alfalfa meal	200	200
Meat scraps	100	100
Salt	20	20
Limestone	30	60
Bonemeal	40	None
Phosphoric acid	None	25
Vitamin premix	10	10
Total	2000	2015

to small particles tied up with organic and mineral ingredient dust.

Test Strip Weight Change. The rate of weight change in the test metal strips was calculated from the formula: $533 \times \text{weight loss in grams} \times 1000$, divided by the test strip density (grams per cc.) \times area in square inches \times hours exposed = mils per year. Evaluation of rates into categories may be shown by the following guide:

Class A. 0 to 5 mils per year corrosion rate; materials suitable for the critical parts where only slight dimensional change can be tolerated.

Class B. 5 to 50 mils per year; materials in common use for noncritical parts where some corrosion can be tolerated.

Class C. Over 50 mils per year. If the products of corrosion are not detrimental to the final product and the

corrosion rate is not excessively above the 50 mils per year rate, the metal should be satisfactory.

All weight changes were considered "weight loss" from the action of basal ration and "corrosion loss" from the action of acid-containing rations. The corrosion loss was not corrected for abrasion loss as is sometimes done to measure the additional effects of corrosion as separate from those of abrasion.

New test strips attached to the mixer sides showed no weight change except, as shown in Table III, opposite the shaft on the "up" side of the feed movement. Here mild weight loss occurred, well within the range of Class B materials. Low-level acid addition continued for 11 hours (actually 110 500-pound mixes with a 6-minute cycle per mix) showed some corrosion at all positions not significantly out of the Class B range.

Following the use of acid, a period of mixing with basal ration showed weight loss at all positions. Examination of test strips showed varying amounts of pitting from previous acid contact, probably accounting for much of the weight change under conditions of basal ration mixing, following the use of acid. This softened metal was not removed by the mild detergent wash and the drying preparatory to weighing. Hereafter, all strips subjected to acid mixing were buffered on a wire wheel before they were put back into use. These test strips before buffing was adopted are designated in Table III as "Basal (ff acid)."

Test strips secured to the lead side of the ribbon agitators showed mild weight loss with basal ration mixing. Strips on the trail side of the agitators remained practically unchanged. Under conditions of low-level acid addition, lead side strips showed a calculated rate of corrosion not much above 50, which is suitable for this type of application. The trial sides of the agitators subjected to acid mixing showed more weight loss than under basal ration mixing conditions. After the feed left the mixer there was no place in the conveying equipment where corrosion was noticeable above the normal abrasion weight loss, until the feed reached the pellet mill. Here in the conditioner chamber, where steam is introduced as an aid in pelleting, excessive corrosion was noted with basal ration operations. Under encrusted feed on the side walls, large rust flakes were stripped off to make a level setting for test strips. The lead side of the mixing paddles which forced the soft feed to the outlet were shiny and not caked with feed. It was noted that caked feed on the paddles was drier than that on the conditioner sides.

Dry Acid Premix. Small batches of acid, limestone, and soybean oil meal were prepared in laboratory mixing equipment, but with a considerable increase in the particles retained on a

Table II. Amount of Ration, in Grams, Mixed with and without Acid, Retained on Tyler Sieves

(Each number an average of five samples)

Test No.	Tyler Sieve Sizes							
	Without Acid				With Acid			
	9	12	24	Pan	9	12	24	Pan
1	1	2	39	159	1	2	40	157.5
2	1	2	38	160	1	2	42.5	155.5
3	1	2	39	159	1	2	43	155
4	1	2	45	154	1	2	42.5	155.5
5	1	2.5	49	148.5	1	2	48	150
6	1	2	46	153	1	2	48.5	150.5
7					1	2	48.5	150.5
8					1	2.5	51	146.5
9					1	2.5	53	144
10						2	47	151.5
Av.	1	2.1	42.7	155.6	1	2.1	46.4	151.7

Table III. Test Strip Weight Changes and Calculated Rate of Corrosion

Position of Strip	Treatment	Time, Hours	Weight Change, G.	Rate, Mils/Yr.
Mixer up side Shaft level	Basal	38.2	-0.0083	8
	Low-level acid	11	-0.0146	48
	Basal (ff acid)	13	-0.0071	20
	High-level acid	4	-0.0266	160
Top spiral level	Basal	38.2		0
	Low-level acid	11	-0.0121	40
	Basal (ff acid)	13	-0.0022	6.1
	High-level acid	4	-0.0270	24.3
Mixer down side Shaft level	Basal	38.2	-0.0009	1
	Low-level acid	11	-0.0241	79
	Basal (ff acid)	13	-0.0184	51
	High-level acid	4	-0.0093	84
Top spiral level	Basal	38.2	-0.0001	0
	Low-level acid	11	-0.0041	13
	Basal (ff acid)	13	-0.0013	3.6
	High-level acid	4	-0.0031	28
Outer spiral, discharge end of mixer Lead side	Basal	38.2	-0.0179	17
	Low-level acid	11	-0.0159	52
	High-level acid	4	-0.0077	69
	Trail side	Basal	38.2	-0.0016
Low-level acid		9.5	-0.0028	11
High-level acid		4	-0.0144	130
Outer spiral, oppos. discharge end Lead side	Basal	38.2	-0.0111	10.5
	Low-level acid	11	-0.0264	86
	High-level acid	4	-0.0167	150
	Trail side	Basal	38.2	
Low-level acid		11	-0.0034	12
High-level acid		4	-0.0146	132
Reverse spiral, center of mixer Lead side	Basal	38.2		0
	Low-level acid	11	-0.0092	30
	High-level acid	4	-0.0019	18
	Trail side	Basal	38.2	
Low-level acid		11	-0.0036	11.5
High-level acid		4	-0.0003	3
Mixer discharge spout	Basal	10	-0.0015	5.4
Elevator discharge spout	Basal	5	-0.0017	12
	Low-level acid	6	-0.0022	13
Elevator cup	Basal	9.4	-0.0049	19
	Low-level acid	5.0	-0.0018	13
Pellet mill conditioner	Basal	26	-0.2596	361
	Low-level acid	5	-0.1078	776
	Dry acid premix	4	-0.0280	280
	Lead side mixer paddle	Basal	31	-0.0808
Low-level acid		4	-0.0258	233
Dry acid premix		8	-0.0160	72
Trail side mixer paddle	Basal	31	-0.1586	185
	Low-level acid	4	-0.0336	302
	Dry acid premix	8	-0.0440	198

No. 9 Tyler screen. After evidence of only mild corrosion in the mixing equipment when using low-level acid additions, high-level acid premixes were tried containing an analyzed total phosphorus content of up to 10%. Table III shows the corrosion resulting from four batches containing 21 to 35% liquid acid carried on limestone and soybean oil meal. Mixing time was extended beyond that necessary to add the acid in order to subject the test strips to acid mixtures for a total of 4 hours and to encourage the chemical reaction of acid and limestone. However, the actual time needed to discharge the acid through the nozzles, recharge the acid feed tank, and finish a batch, was over 3 hours for four batches.

The formulas for dry acid premix (Table IV), show that in batches 3 and 4, acid was well in excess of the ability of the limestone present to neutralize it, if proper conditions existed for a chemical reaction to be carried to completion. Completeness of reaction (Table V), based on the measurement of limestone carbon dioxide not evolved, showed that during mixing and 10 minutes after, 40% or more of the theoretical reaction had taken place. Calculated final bath weights were based on the completeness of the reaction at 8 weeks. Weight loss of the batch was calculated according to the formula: % CaCO₃ in limestone × pounds limestone in batch × % reaction × 44, 100. High-acid premixes varied little in texture from the soybean oil meal used as a carrier (Table VI).

Pelleting. The reported pelleting data were an average of five separate runs of 1 ton each of basal ration, followed by a ton or more containing liquid acid, and two runs containing dry acid premix. Before recording any data, one mixer load of 500 pounds basal ratio was pelleted to bring the equipment to normal temperature conditions. Steam was introduced into the pellet mill by a Hancock flow control valve which had been calibrated to relate turns of the wheel to pounds of steam per hour. All pelleted feeds were crumbled and the fines returned to the mill for repelleting were collected at the sifter by removing the usual spouting and sacking off the "throughs" of the sifter screen, as they were produced. Pellet hardness was measured on a Stokes hardness tester.

A power panel wired to the pellet mill motor indicated operating voltage, amperes, watts, and power factor. In addition, a four-digit counter recorded disk revolutions of a watt-hour meter. The watt-hour tally was used in all cases to calculate kilowatt-hours of energy used per ton of pelleted feed.

Pelleting specifications for basal rations and those containing liquid acid and dry acid premix are shown in Table VII. Pelleting time in minutes per ton included repelleting the fines returned to the mill during continuous operation.

Table IV. Formulas for Dry Acid Premix and Calculated Total Weight after 8 Weeks' Storage

Batch No.	Limestone		Soybean Oil Meal		75% Acid		85% Acid		Mixing Weight, Lb.	Final Weight, Lb.
	%	Lb.	%	Lb.	%	Lb.	%	Lb.		
1	18.7	62.4	60.0	200	21.3	71	333.4	315.6
2	17.8	62.4	57.1	200	25.0	87.6	350	334
3	16.6	62.4	53.4	200	30.0	112.6	375	353.5
4	16.4	84.0	49.6	270	35	190	544	515

Steam pressure with the flow control valve closed was adjusted by a regulatory valve to 90 p.s.i.g. and the drop in pressure noted was due to the opening of the valve to the pellet mill conditioner chamber. The ratchet drive volumetric feeder was set as high as possible without exceeding full load rated current.

Discussion

Specifications for good acid distribution in feeds required the use of flat nozzles which produced an atomizing, single plane type of spray, discharging about 10 inches from the rapidly moving feed, sufficiently dense to prevent penetration of acid to the metal parts of the mixer. Poor acid distribution was associated with an increase in the weight of particles retained on a Tyler No. 9 screen. These large particles contained an excess of liquid acid, as shown by low pH and caking to mixing equipment.

With each nozzle spraying 0.17 gallon per minute at 30 p.s.i.g., the four in use allowed the total weight of low-level acid addition to be made in less than 1 minute. Insufficient feed in the mixer at the time of acid addition or the direction of liquid acid into "dead" areas or onto equipment, where it was picked up by feed, not only caused corrosion in excess of that found with good distribution, but allowed balling up of acid and feed material. Where the trail side of the spirals or low-friction areas of the mixer were wetted with acid, as occurred with poor distribution, subsequent mixing of the basal ration caused corrosion in excess of that found where there was no previous history of acid use. Acid may have softened the metal, causing it to be easily eroded by mild feed friction.

No loss of acid vapor from spray nozzles was noticed, even when the dust-collecting system was not in use. Also, with low-level acid addition, no signi-

Table V. Completeness of Reaction Immediately after Mixing Dry Acid Premix and after 8 Weeks' Storage

Batch No.	10	25	48	8
	Min., %	Hr., %	Hr., %	Weeks, %
1	41.9	42.1	46.0	66
2	40.6	51.9	47.1	59
3	41.0	66.4	58.3	80
4	...	65.4	54.7	80

ficant weight change was observed between material going into the mixer and that sacked off after mixing and conveying to a sack-off bin. It may have been that acid was absorbed by the bulk of the feed material, such as grains and protein concentrates, to be buffered and dried beyond the point of a chemical reaction with limestone. Undoubtedly, the reaction was slowed by the absence of water and a delayed, continuous reaction may take place confined to small balls of acid and feed material where the limestone was intimately mixed. This was noted in the high-level acid mixture where a continuing reaction took place, aggravated by agitation and aeration of the premix material.

Cleaned metal test strips were secured at various positions in the mixing, conveying, and pelleting. When feed was manufactured between tests for weight loss, it was always difficult to locate previous holes in the equipment where strips had been bolted. A few places in the equipment remained shiny and clean, and these, of course, were subject to most abrasion. The step of taking off and replacing clean strips may have resulted in more of a weight change than would have been found under usual operating conditions where a build-up of material is allowed on metal surfaces. In fact, data shown may be considered as extreme for the conditions used. This would not be true of the pellet mill

Table VI. Tyler Sieve Analysis and pH 1% Slurry Dry Acid Premix

Batch No.	Over Tyler No. 10,		Over Tyler No. 16,		Over Tyler No. 20,		Through Tyler No. 20,	
	%	pH	%	pH	%	pH	%	pH
1	0.26	3.7	12.6	5.0	22.6	5.0	64.8	4.75
2	0.12	3.7	10.0	4.7	19.0	4.7	70.6	4.60
3	0.12	3.6	11.0	4.2	21.6	4.0	68.0	3.80
4	0.5	2.7	6.0	3.4	18.0	3.3	75.6	3.30
Soybean oil meal	0.3	6.0			17.12	6.2	82.7	6.2

conditioner chamber, where caked feed never dries and a continuous corrosion is taking place, aggravated by the high temperature as well as moisture.

It was difficult to follow a basal ration in the pellet mill, with one containing liquid acid and to maintain the same mill settings established by the previous run. The first obvious change needed was the reduction of steam intake to prevent choking up of the pellet die. With less steam going into the soft feed, the feeder setting had to be backed off to prevent the wall ammeter from reading beyond a safe amperage for the motor. If the same settings were used, constant attention would be necessary to prevent either a choke-up of the die or an overheating of the motor. When dry acid premix was used in the formulation, no change from the basal ration was made in the pellet mill operations and the only indication of the presence of acid was a slight increase in kilowatt-hours per ton of feed produced.

The increase in pellet hardness in acid feeds was considered significant. The practical value of this was observed in a reduction of per cent fines brought back to be repelleted. The reduced amount of fines developed from pelleted, crumbled acid feeds did not entirely compensate for the lower feeder setting during the pellet operation. Because pellet hardness depends somewhat on moisture content, it is possible that the reduced moisture added to acid feeds resulted in a

Table VII. Production Data when Pelletting Basal, Liquid Acid, Dry Acid Mix Ratios

	Basal Ration	Liquid Acid	Dry Acid Mix
Mixed, pelleted	5	5.8	2.25
Pelleting time, min./ton	33.2	34.6	33.3
Kw.-hr./ton	7.93	9.77	8.86
Steam, p.s.i.g.	48	60	48
Steam flow valve	2.8	1.8	2.8
Pellet feeder setting	35	32	35
Fines returned to mill, %	22	17	No data
Pellet hardness	10.0	10.5	9.9
pH soft feed, 1% slurry	6.2	5.6	5.9
Water added, as steam, %	5.2	4.5	5.2

drier, harder pellet coming from the cooler. On the other hand, liquid (commonly water or steam) is used to bind soft feed material into a hard, compressed form as well as to lubricate the material and increase the pelleting rate. The total liquid added, acid, and steam, was larger in the acid feeds than in the basal ration, which may explain the hardness of the acid-containing pellets.

Undoubtedly, the use of a high-level acid premix, dry and free-flowing with completeness of acid-limestone reaction nearly 100%, would enable mixing, conveying, and pelleting operations to proceed as when using any other dry phosphate compound. A small continuous premixing line using stainless steel equipment could be used to feed phosphoric acid into rations, with adjusted formulas to allow for the composition of the premix.

Acknowledgment

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NIACIN DETERMINATION

Chemical Method for Estimation of Niacin in Poultry Feeds and Premixes

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The need for a chemical method for the routine determination of niacin in biological materials has been evident for some time. A suitable method involving initial purification of extracts followed by colorimetry is presented. Recoveries of niacin averaged 98.6%. Values found by this method agreed closely with those determined by microbiological assay. The procedure can be adapted to the routine determination of niacin in mixed feeds or premixes as part of a feed control program. With suitable modifications, it may also be used to determine niacin in a variety of feed ingredients.

A SIMPLE chemical method for the routine determination of niacin in manufactured feeds and premixes has been needed for some time. Among the numerous methods proposed for estimation of niacin in biological material (1, 5, 6, 9, 10), that of Chitre and Desai (4) appeared most adaptable for routine use. These authors used the potassium permanganate decolorization procedure of Krehl, Strong, and Elvehjem (8) to purify the extracts, and Koenig's reaction of α - or γ -unsubstituted pyridine derivatives with cyanogen bro-

midate and an aromatic amine to give a color reaction (7). Aniline hydrochloride was the aromatic amine used rather than Swaminathan's aniline (11).

The method presented herein is a modification of the above procedure, and has proved very satisfactory for routine determination of niacin or niacinamide in manufactured feed and premixes.

Analytical Procedure and Results

Reagents. A 3% solution is prepared by dissolving cyanogen bromide (Eastman Kodak) crystals in distilled

water, or by adding a 10% solution of potassium or sodium cyanide, dropwise, to a cold saturated solution of bromine until the solution is just decolorized. The filtered solution will keep for 4 to 5 months if stored in the cold, in an amber-colored bottle.

Aniline Hydrochloride. To 40 ml. of redistilled aniline in an ice bath, under a hood, 100 to 130 ml. of concentrated hydrochloric acid are added, a few milliliters at a time with constant stirring, until the creamy white precipitate is completely formed. The pre-