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POTENTIAL FERTILIZERS FOR DEVELOPING COUNTRIES

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This report presents a brief summary of tests recently made or in progress for production of several potential fertilizer materials that may have application in developing countries. Such materials include urea-phosphate rock, urea-single superphosphate based materials, urea-ammonium sulfate, granulated rock phosphate and improved urea. Emphasis is placed on the use of urea since it is now and will continue to be the most important nitrogen source in developing countries. Tests thus far are promising for development of a coating material which will improve the physical characteristics of urea used under tropical conditions.

The fertilizer products have been produced in laboratory equipment and pilot plants only to demonstrate the technical feasibility of the process, to identify variables and their effects, provide information for further scale up, and to produce sufficient quantities of experimental products for agronomic testing. Data in this report should not be considered necessarily as optimum values for variables. Operating data given are typical values obtained in laboratory and pilot-plant work.

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Summary

Potential Fertilizers for DEVELOPING COUNTRIES..

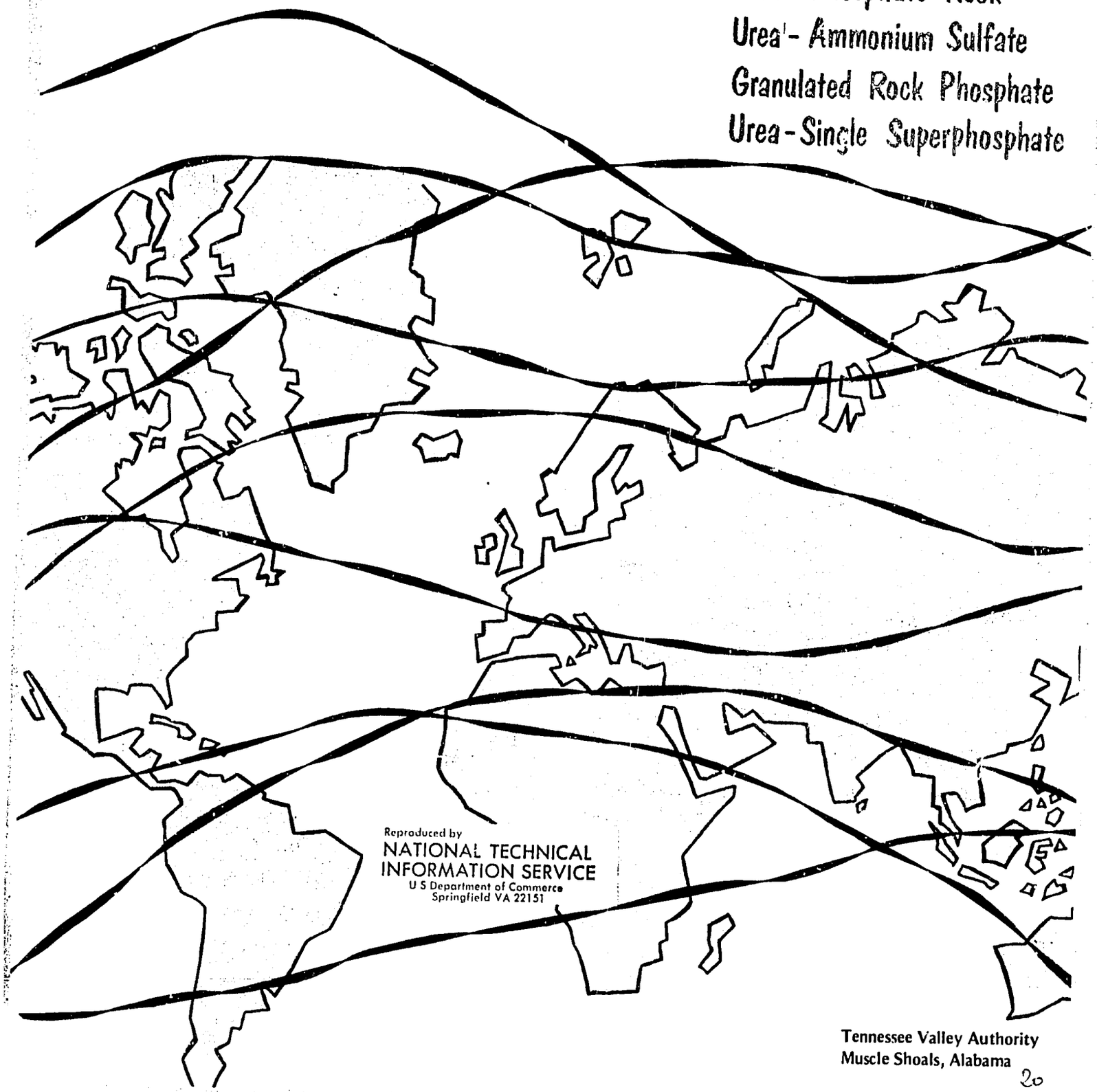
Improved Urea

Urea - Phosphate Rock

Urea - Ammonium Sulfate

Granulated Rock Phosphate

Urea - Single Superphosphate



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LIST OF ABBREVIATIONS

mt/hr	– metric ton per hour
st/hr	– short ton per hour
kg/hr	– kilogram per hour
lb/hr	– pound per hour
kg/mt	– kilogram per metric ton
lb/st	– pound per short ton
cm	– centimeter
in	– inch
kg/m^3	– kilogram per cubic meter
lb/ft^3	– pound per cubic foot
$kg/hr/mm^2$	– kilogram per hour per square millimeter
$lb/hr/mm^2$	– pound per hour per square millimeter
rpm	– revolutions per minute
G	– centrifugal force; $= 14.2 \times 10^{-6} \times n^2$ (speed of rotation, rpm) $\times d_b$ (diameter of bowl in inches)
mg/cm^2	– milligram per square centimeter

Prepared for

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by PASA TA(QA) 6-69

National Fertilizer Development Center
Tennessee Valley Authority
Muscle Shoals, Alabama 35660

INTRODUCTION

The Tennessee Valley Authority (TVA) in cooperation with the United States Agency for International Development (USAID) is conducting tests for production and evaluation of fertilizer products that are of interest primarily for developing countries. Such materials include urea-phosphate rock, urea-single superphosphate based materials, urea-ammonium sulfate, granulated rock phosphate, and improved urea. Emphasis is placed on the use of urea since it is now and will continue to be the most important nitrogen source in developing countries. Demonstration of ways to improve its physical properties and ways to use it with certain other materials might further enhance the role of urea. Even now bulk urea is moving into tropical areas, a practice once considered quite risky. The increased availability of bulk urea and urea production facilities in developing countries will allow a wide range of urea products to be made.

OBJECTIVES AND SCOPE OF WORK

This report presents a brief summary of tests recently made or in progress for production of several potential fertilizer materials that may have application in developing countries. In many cases further work is planned; thus, this may be considered as a progress report. The contribution and scope of future work will depend upon interest and availability of funds. Since many of the products have applicability primarily in developing countries support for experimentation has come primarily from non-TVA sources.

TVA does not normally design, fabricate, or construct full-scale plants or plant equipment, and the processes described herein have not been developed to a commercial scale. The products were produced in laboratory equipment and pilot plants only to demonstrate the technical feasibility of the process, to identify variables and their effects, provide information for further scale up, and to produce sufficient quantities of experimental products for agronomic testing. TVA is willing to cooperate with and share the data available with interested parties to enable experienced groups to utilize the information in carrying out further testing or for consideration in preliminary design for process development. Data in this report should be helpful in these respects, but should not be considered necessarily as optimum values for variables. Also, the flow diagrams are suggested routes to production of the products, but are not meant to limit the choice or use of other equipment and schemes. Operating data given are typical values obtained in laboratory and pilot-plant work.

PRODUCTS AND PROCESS DESCRIPTIONS

Raw Materials Used in the Tests

Chemical and physical analyses data for the raw materials used in preparation of experimental materials described in this report are given in table 1. Most of these materials were obtained from local suppliers for use in the experimental program. These are typical materials which should be available or equivalent materials could be obtained in many developing countries. The phosphate rock was usually obtained ground to the size used to produce superphosphate. Since no concentrated urea solution is available at TVA, urea prills were melted when needed to simulate the concentrated solution that would be obtained from the concentrator in a urea process.

Table 1. Typical chemical and physical analyses of raw materials used[a]

	Normal superphosphate (0-20-0)	Phosphate rock[b] (0-30-0)	Urea prills (46-0-0)	Ammonium phosphate (13-52-0)	Diammonium phosphate (18-46-0)	Potassium chloride (0-0-60)
Chemical analysis, %						
Total N	–	–	46.3	13.1	17.8	–
Biuret	–	–	0.78	–	–	–
Total P_2O_5	19.3	29.6	–	52.2	46.7	–
Citrate-insoluble P_2O_5	1.0	–	–	0.13	0.66	–
Water-soluble P_2O_5	16.5	–	–	45.7	39.4	–
Free-acid P_2O_5	2.1	–	–	–	–	–
K_2O	–	–	–	–	–	61.2
H_2O (AOAC method)	6.5	2.0[c]	2.0[c]	1.95	0.98	0.07
CaO	–	47.7	–	–	–	–
Screen analysis (Tyler), %						
+6 mesh	10.2	–	0.2	0.2	0.0	0.1
-6 +8 mesh	8.3	–	1.3	18.9	20.7	0.2
-8 +10 mesh	8.8	–	33.5	67.2	70.7	0.4
-10 +16 mesh	12.3	–	63.7	13.0	8.4	5.4
-16 +20 mesh	4.4	–	0.6	0.4	0.2	8.7
-20 +28 mesh	6.6	–	0.4	0.2	0.0	21.8
-28 mesh	49.4	–	0.3	0.1	0.0	63.4
-35 +80 mesh	–	2.3	–	–	–	–
-80 +100 mesh	–	3.2	–	–	–	–
-100 +200 mesh	–	19.3	–	–	–	–
-200 + 325 mesh	–	18.6	–	–	–	–
-325 mesh	–	56.6	–	–	–	–

[a]Raw materials obtained from local suppliers.
[b]Uncalcined North Carolina, USA, phosphate rock.
[c]Karl Fischer moisture.

Granulated Urea-Phosphate Rock

Granulated urea-phosphate rock is a homogenous mixture of urea and fine-ground (75% -200 Tyler mesh) phosphate rock. Urea provides the granulating medium for binding together the small rock particles. In order for the rock to be available to crops, it must be either applied as a fine grind or rapidly converted to its original fine-ground form. This is accomplished by the rapid dissolution of the urea in water which allows fast disintegration of the granules to provide fine-ground material. Obviously, the handling, storage, transport, and application characteristics of granulated materials are preferred over a dusty material such as ground rock. Two grades of materials were produced, 26-13-0 and 18-18-0.

Process—Figure 1 shows one process used for production of the urea-phosphate rock granules in the TVA pilot plant. Tests have been conducted in two types of granulation equipment, a pan-type and rotary drum granulator. Results were superior when the pan method was used.

Concentrated urea solution of 98 to 99.5% concentration is forced by pressure pump through spray nozzles onto a moving bed of solids in a pan granulator. Phosphate rock is ground and fed along with recycle fines to the granulator. Since the urea concentration is relatively high, no dryer is required. Material from the granulator goes to a rotary countercurrent cooler. After cooling, material is screened to separate oversize and fines from the product. Fines are recycled to the granulator as a heat sink and as nuclei for granulation. Product is conditioned, stored, or bagged. Alternatively for locations where concentrated urea solution is not available, urea prills can be melted and the melt handled in the same way as the concentrated urea solution. At TVA, urea prills were melted in a steam-heater melter. Unconditioned prills were used.

Tests were also made in which granulation was carried out in a rotary drum granulator. Granulation was poor in these tests and a large amount of fines was collected in the cyclones. Also, the recycle stream consisted of small dusty particles. Based on data available, the pan granulator appeared to be superior to a drum granulator for urea-phosphate rock.

Typical Operating Conditions—Table 2 gives typical operating conditions in the pilot plant for pan granulation

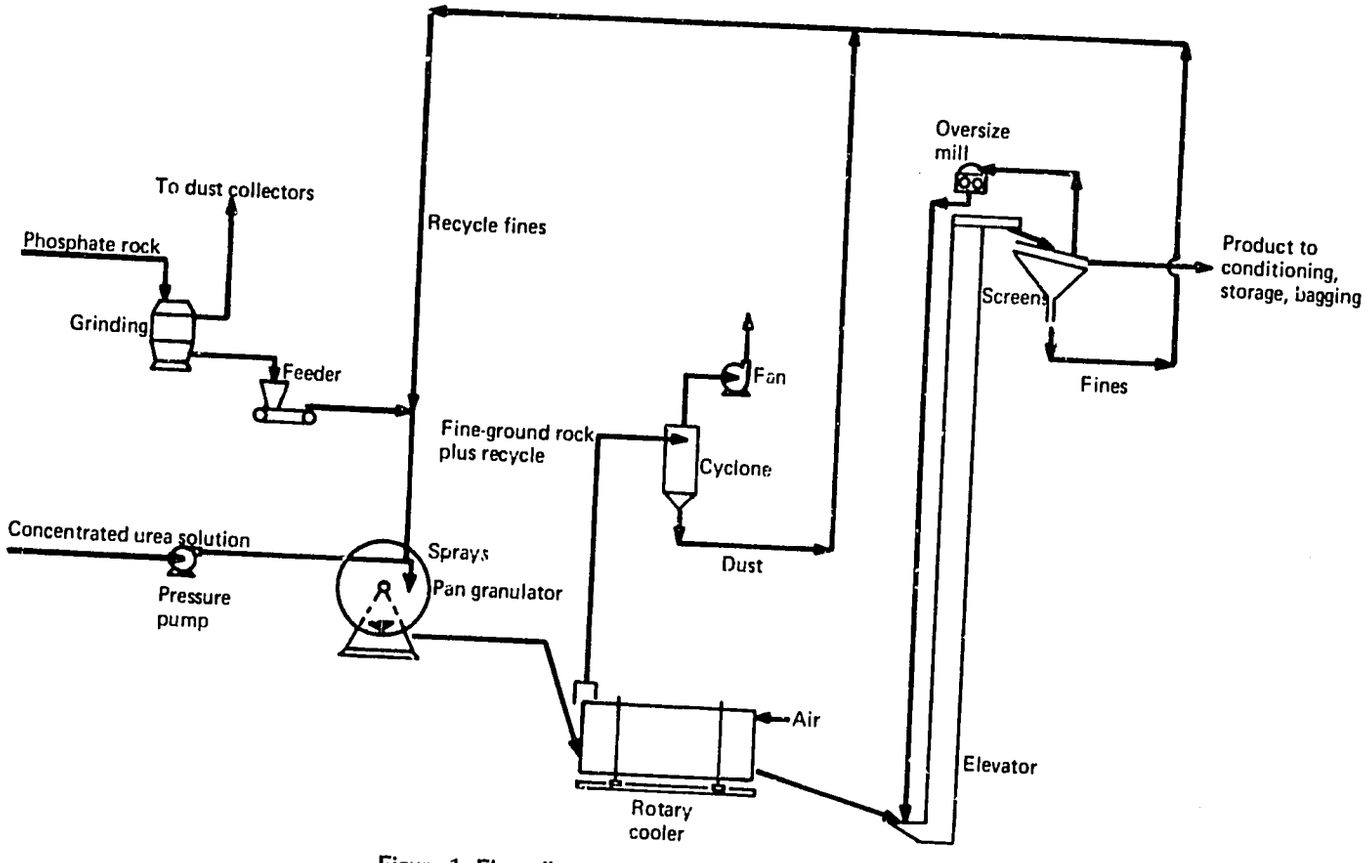

Figure 1. Flow diagram for urea-phosphate rock granules

Table 2. Operating conditions for pan[a] granulation of urea-phosphate rock

Nominal grade	26-13-0	18-18-0
Nominal production rate, mt/hr (st/hr)	0.68(0.75)	0.998(1.1)
Urea feed rate to melter, kg/hr (lb/hr)	389(855)	389(855)
Phosphate rock feed rate, kg/hr (lb/hr)	295(48)	589(1296)
Recycle fines to pan granulator, kg/hr (lb/hr)	491(1080)	255(560)
Recycle ratio, kg/kg (lb/lb) of product	0.72	0.26
Operating temperatures, °C (°F)		
Urea solution to sprays	152(306)	150(303)
Material in granulator	112(235)	106(224)
Cooler discharge	83(182)	91(196)
Screened product	74(165)	85(186)
Recycle fines	65(149)	72(161)
Number of sprays to pan granulator	3	3

[a]Pan granulator having a diameter of 38 inches.

of urea-phosphate rock. The nominal production rate ranged from 0.63 to 0.91 mt/hr (0.7 to about 1 st/hr). The urea feed rate was kept constant at 386 kg/hr (850 lb/hr). The rock feed rate was varied to obtain the desired grade of product, 295 to 591 kg/hr (650 to 1300 lb/hr). The urea solution was kept at about 151° C (305° F) with a concentration of about 99%. To compensate for high heat losses in the pilot plant, some heat was added to the cooler in these tests. The temperature of the rolling bed in the granulator ranged from 107° to 112°C (224° to 235°F).

Results—The data in table 3 show that urea-phosphate rock can be successfully granulated in the pan-type granulator. Essentially all of the screened product was larger than 16-mesh (Tyler). The nutrient content of the products was

Table 3. Results of tests for pan granulation of urea-rock phosphate

Screen analysis (Tyler) %	26-13-0	18-18-0
Granulator product		
+4 mesh	1.1	0.0
-4 +6 mesh	3.3	2.6
-6 +8 mesh	16.9	26.3
-8 +10 mesh	33.[illegible]	37.2
-10 +12 mesh	15.9	11.8
-12 +16 mesh	17.8	9.1
-16 +28 mesh	6.4	2.6
-28 mesh	5.2	10.2
Screened product		
+4 mesh	0.0	0.0
-4 +6 mesh	0.6	0.4
-6 +8 mesh	30.4	25.9
-8 +10 mesh	44.1	45.7
-10 +12 mesh	15.1	15.3
-12 +16 mesh	9.5	11.2
-16 +28 mesh	0.1	1.3
-28 mesh	0.2	0.2
Chemical analysis[a], *wt %*		
Total N	26.4	18.6
Biuret	1.2	0.7
Total P_2O_5	13.3	18.2
H_2O (Karl Fischer)	0.12	0.21

[a]Unconditioned basis.

on grade with no conditioner. The biuret content ranged from 0.7 to 1.2%. This was relatively low, but should be even less if a concentrated urea solution is available. Some small amount of biuret formed during melting of the urea because of poor design of the melter. The granules were well rounded and appeared to consist of a core of urea with a coating of phosphate rock.

The final screened products were coated with 2% celatom for conditioning and both conditioned and unconditioned products were stored up to 9 months. The products stored quite well for 9 months even when unconditioned. Moisture content of the materials was about 0.1-0.2%. No data are available to indicate bulk storage characteristics or storage under tropical conditions.

Applications—Some developing countries use phosphate rock for direct application, and farmer complaints have been received for handling and application of the fine-ground rock. Various methods are being sought to granulate the rock. The use of urea would be a very convenient way to achieve granulation.

Further tests are needed on ways to granulate urea-phosphate rock. For example, the use of crushed urea prills and fine-ground rock with steam granulation in a rotary drum should be tested. Also, a pugmill might be used for this process. Inclusion of other materials such as a small quantity of water-soluble phosphate for early crop response might be helpful.

The products are being tested agronomically for rice and much interest in such products is being shown in Indonesia. The product should have good potential for Brazil. Almost any area having acidic soils could utilize such a product. However, the agronomic effectiveness of ground rock is dependent on its reactivity and TVA is evaluating various rocks in this respect. A report is available which discusses the relative reactivity of selected rock phosphates.

Granulated Materials Containing Urea-Single Superphosphate without Ammoniation

Single superphosphate (SSP, 0-20-0) is often a very economical source of phosphate in many developing countries, and also it is a source of sulfur. In many cases, it is a good outlet for utilization of byproduct sulfuric acid. However, due to the relatively low analysis (20% P_2O_5) it is difficult to bag and ship the material over long distances and remain competitive with higher analysis materials such as diammonium phosphate (DAP, 18-46-0), monoammonium phosphate (MAP, 11-55-0), or triple superphosphate (TSP, 0-46-0). TVA in cooperation with AID has been testing the production of urea-single superphosphate based compound NPK granular products for these situations. Many countries market phosphates on the basis of water solubility. When single superphosphate is ammoniated, the water solubility is significantly reduced. Therefore, the process studied involved little or no ammoniation.

Generally mixtures of urea-single superphosphates have been avoided because of the possibility of formation of an adduct which releases water and renders these fertilizers tacky and wet during processing. Some tests were made in bench-scale equipment prior to testing in larger-scale pilot plant equipment. Some work has been done in India on this process. Also, some products are made in Great Britain and Japan containing urea and unammoniated or lightly ammoniated single superphosphate.

Process—The flow diagram for production of urea-single superphosphate based granules is shown in figure 2. No tests have been made without supplemental sources of other phosphates such as MAP, DAP, and SSP. In order to make relatively high analysis grades, it is necessary to supplement the single superphosphate with other phosphate materials such as diammonium phosphate or monoammonium phosphate. In some of the formulations, crystalline ammonium sulfate was used. Urea was used either in the form of microprills (about 80% -16 mesh) or standard size prills. Potash was added as standard red KCl (about 83% -20 mesh).

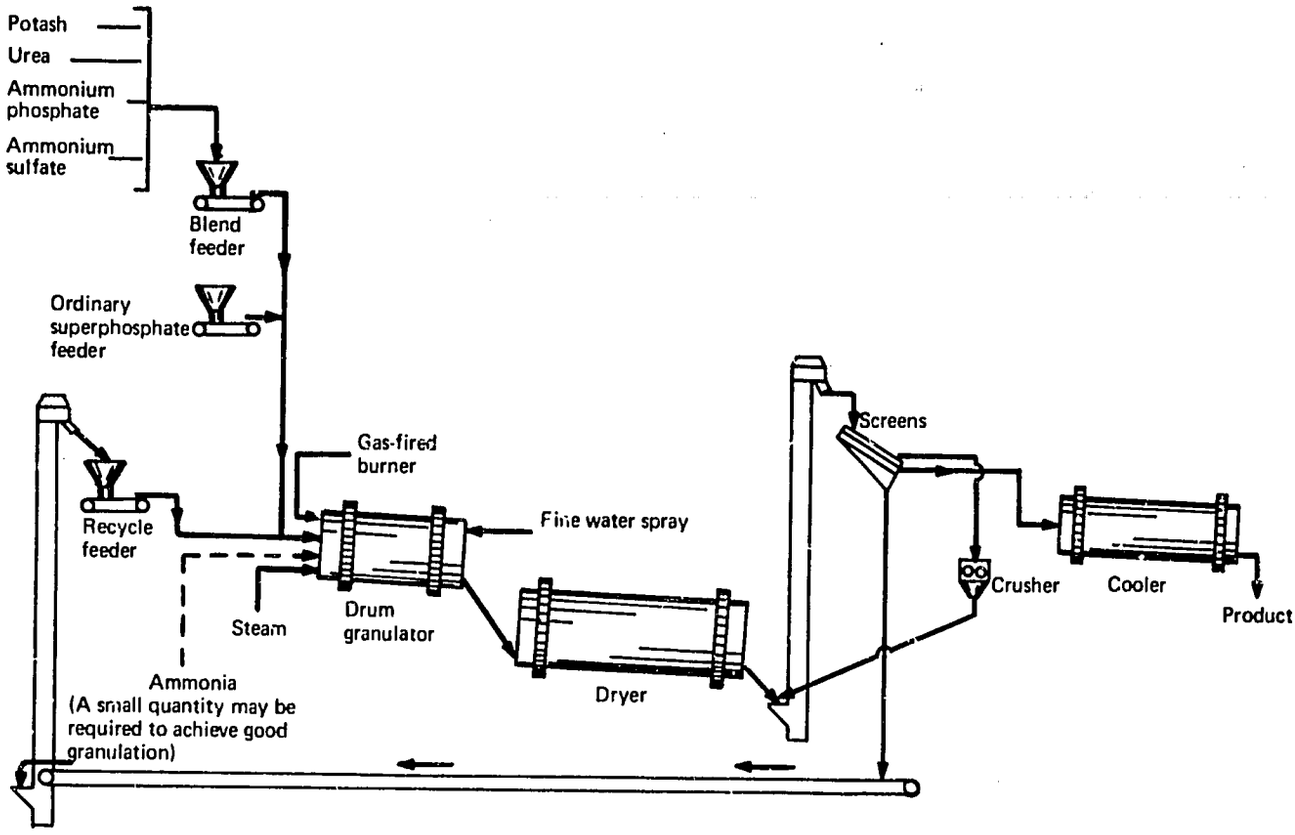

Figure 2. Flow diagram for urea single superphosphate based granulated products

Basically, granulation is achieved by addition of external heat or by the addition of water or steam to the rolling bed of particles.

In the range of 54° to 82°C (130° to 180°F) the reaction to form an adduct occurs which releases water to aid the granulation. The reaction can be represented by the following equation:

$$4\,CO(NH_2)_2 + Ca(H_2PO_4)_2 \cdot H_2O \rightleftharpoons Ca(H_2PO_4)_2 \cdot 4CO(NH_2)_2 + H_2O$$

Solid raw materials, such as urea, ammonium phosphates (MAP or DAP), and ammonium sulfate, are fed to a mixer to blend uniformly. This is not necessary but was convenient to assure more uniform feeding. After mixing, the material is metered to the drum granulator with a variable-speed feeder. The fine-size single superphosphate is fed with a separate disc feeder and recycle fines are fed with a third feeder. All of the solid materials feed to a common chute before entering the granulator so that some mixing can occur here. Heat is supplied through a gas-fired burner which uses acetylene or natural gas and air. The burner consisted of essentially a drilled-pipe sparger and is installed above the bed and near the feed end of the granulator. It is located just downstream of the steam sparger (underbed) and flames impinge directly onto the moving bed. Near the outlet or discharge end of the granulator an air-atomizing water nozzle is installed above the bed to allow touchup of the moisture content. In the pilot plant, no cooler was used after the dryer and material was screened hot. It is advantageous to recycle hot material to conserve heat. The screened product is cooled prior to bulk storage or bagging.

The process has been studied in three sizes of equipment starting with a laboratory-scale rotary drum of 0.9 kg (2 lb) capacity, a continuous small-scale pilot plant of 45 kg/hr (100 lb/hr), and a continuous pilot plant of 0.90 mt/hr (1 st/hr).

Throughout the testing period, changes were made in equipment and procedure in an attempt to obtain optimum granulation.

Typical Operating Conditions—Table 4 presents data obtained from the small-scale pilot plant during production of 14-14-14 and 10-20-10 grades, using supplemental sources of phosphate such as mono- and diammonium phosphates. Urea was used in the form of microprills. The granulator temperature ranged from 65° to 83°C (149° to 182°F). In the 14-14-14 material, the urea content was 23% and about 13% in the 10-20-10 grade. A higher proportion of single superphosphate was used in the

Table 4. Operating conditions for production of granular urea-single superphosphate products[a] (small-scale pilot-plant)

Nominal grade	14-14-14			10-20-10
Test no.	1	2	3	4
Formulation, kg/mt (lb/st)				
Ammonium sulfate	71.5(143)	96(192)	40(80)	–(–)
Ammonium phosphate (13-52-0)[b]	146.5(293)	–(–)	–(–)	–(–)
Ammonium phosphate (11-55-0)[c]	–(–)	148.5(297)	–(–)	–(–)
Ammonium phosphate (18-46-0)[d]	–(–)	–(–)	181.5(363)	227(454)
KCl	233.5(467)	233.5(467)	234.5(469)	166.5(333)
Single superphosphate	318.5(637)	292.0(584)	314.0(628)	477.5(955)
Urea	230.0(460)	230.0(460)	230.0(460)	129.0(258)
Weight percent urea in formulation	23.0	23.0	23.0	12.9
Feed rates, kg/hr (lb/hr)				
Blend	30.9(68)	30.5(67)	31.8(70)	24.1(53)
Single superphosphate	14.5(32)	12.7(28)	15.5(34)	20.5(45)
Recycle	45.5(100)	32.7(72)	57.9(127.5)	50.0(110)
Steam	0(0)	0.3(0.6)	0.7(1.5)	–(–)
Total input water, % of feed	0.9	2.5	2.0	1.4
Granulator temperature, °C (°F)	83(182)	73(163)	65(149)	76(168)
Recycle ratio, kg/kg (lb/lb) of product	1.0	0.7	1.3	1.1
Dryer-product temperature, °C (°F)	72(162)	68(155)	69(157)	51(124)

[a]Tests in 45.5 kg/hr (100 lb/hr) granulation plant.
[b]Granular ammonium orthophosphate.
[c]Powdered monoammonium phosphate.
[d]Granular diammonium phosphate.

10-20-10 grade. During operation, the recycle ratio was approximately 1 kg/kg (1 lb/lb) of product. Initial studies were made with a 15-15-15 grade which contained 28.5% urea but the liquid phase was excessive. Another formulation having less urea (23% versus 28%) with nitrogen obtained from ammonium sulfate was not good either since the quantity of single superphosphate was too low to obtain good granulation. By increasing the quantity of single superphosphate it was expected that the water solubility would be decreased somewhat. It still should be around 80% or higher for most formulations. With the feed rate of about 45 kg/hr (100 lb/hr), the retention time in the granulator was about 20 minutes.

Data obtained during operation of the larger-scale 0.90 mt/hr (1 st/hr) pilot plant for production of 14-14-14, 12-12-12, 11-11-11, and 10-20-10 grades are shown in table 5. In this equipment operating conditions were more easily controlled and the provision for addition of anhydrous ammonia was available. It should be pointed out that the main objective was to achieve granulation without ammoniation in order to have a high degree of water solubility in the product. The process would be much simpler and less difficult if some ammonia such as anhydrous, aqueous, or ammonia-urea solution could be added to react with the single superphosphate (or MAP) to generate heat. This, of course, reduces the water solubility of the phosphate. In the larger unit, urea was added in the form of standard prills in the range of 13 to 25% of the formula weight. The quantity of phosphate derived from single superphosphate in the formulations ranged from about 30 to 100%. Granulator temperature ranged from a low of 43°C (109°F) to a high of 82°C (179°F). The recycle ratio also had a wide range from 0.3 to 1.3 kg/kg (lb/lb) product.

Results—Typical results from tests in two sizes of pilot plants are shown in table 6. The main information determined from tests in the small pilot plant was that the process requires close control to prevent over or undergranulation and that a high percentage of urea in the formulation should be avoided. Single superphosphate was helpful in achieving good granulation. Some water solubility was lost when diammonium phosphate was used in the formulation due to the reaction with superphosphate. Hydrolysis of urea with formation of ammonia resulted in loss of water soluble P_2O_5. For the 14-14-14 grade, best overall operation was obtained in test 1 in which the ammonium orthophosphate was crushed. Granulation with diammonium phosphate (test 3) was also good. When powdered monoammonium phosphate was used (test 2) very precise control of solids feed, steam flow, and heat input was required to achieve good granulation. Water solubility ranged from 68 to 87% with this grade and was highest when powdered monoammonium phosphate was used as the source of supplemental P_2O_5. For the 10-20-10 grade only diammonium phosphate was used as the source of supplemental phosphate. This grade required about 8% moisture in the granulator at 76°C (168°F) to obtain good

Table 5. Operating conditions for production of granular urea-single superphosphate based products[a] (large-scale pilot-plant)

Nominal grade	14-14-14		12-12-12	11-11-11		10-20-10	
Test no.	1	2	3	4	5	6	7
Feed rate, kg/hr (lb/hr)							
Ammonia, anhydrous	(–)	(–)	(–)	(–)	(3)	(–)	(20)
Urea prills[b]	214.5(472)	169.1(372)	225.5(496)	224.1(493)	151.8(334)	121.8(268)	116.8(257)
Ammonium sulfate	66.8(147)	149.5(329)	(–)	(–)	122.7(270)	45.9(101)	43.9(96)
Ammonium phosphate (13-52-0)[c]	136.8(301)	160.5(353)	(–)	(–)	(–)	215.5(474)	205.9(453)
Ammonium phosphate (18-46-0)[c]	(–)	(–)	29.5(65)	(–)	27.3(60)	(–)	(–)
KCl	218.2(480)	216.4(476)	181.8(400)	171.4(377)	168.2(370)	156.4(344)	149.5(329)
Single superphosphate	300.0(660)	227.3(500)	447.3(984)	525.0(1155)	437.3(962)	369.6(813)	351.8(774)
Recycle	(–)	609.1(1340)	106.4(2340)	300.0(660)	790.9(1740)	818.2(1800)	822.7(1810)
Steam	(–)	(–)	(–)	(–)	(–)	0	0
Water	(–)	2.3(5)	(–)	(–)	(–)	0	0
Wt % urea in formulation	22.9	18.3	25.5	24.3	16.7	13.4	13.3
Total input water, % of feed	2.6	2.1	2.4	3.6	2.5	4.7	4.4
Granulator temperature, °C (°F)	57(134)	70(158)	52(126)	43(109)	82(179)	46(114)	76(169)
Recycle ratio, kg/kg (lb/lb) of product	(–)	0.66	1.27	0.33	0.87	–	–
Recycle temperature, °C (°F)	32(89)	34(94)	41(106)	23(73)	47(116)	51(124)	54(129)
Dryer-product temperature, °C (°F)	62(144)	73(163)	80(176)	54(129)	97(207)	67(152)	81(177)

[a]Tests in 0.90 mt/hr (1 st/hr) granulation plant.
[b]Standard prills.
[c]Granular.

Table 6. Results of tests for production of urea-single superphosphate based granules in pilot plants

Nominal grade	Small-scale[a]				Large-scale[b]						
	14-14-14			10-20-10	14-14-14		12-12-12	11-11-11		10-20-10	
Test no.	1	2	3	4	1	2	3	4	5	6	7
Onsize product data											
Chemical analysis, % by wt											
Total N	13.2	12.9	11.9	10.0	14.8	13.7	18.5	12.5	13.4	12.6	12.3
NH_3-N	–	–	–	3.8	–	–	–	–	–	–	–
Biuret	–	–	–	0.24	–	–	–	–	–	–	–
Total P_2O_5	15.2	14.2	14.4	20.1	16.7	14.8	12.2	11.1	13.2	25.9	24.2
Available P_2O_5	14.7	14.0	13.7	19.0	16.5	14.6	11.8	10.5	12.9	25.5	23.7
Water-soluble P_2O_5	12.4	12.4	9.8	12.6	12.5	12.7	9.2	9.6	8.1	21.9	19.3
Free-acid P_2O_5	0.1	0.3	–	–	0.05	0.02	0.21	1.1	0.04	0.02	0.02
K_2O	14.2	14.9	18.7	11.0	14.3	15.3	6.4	11.8	9.1	9.4	9.5
H_2O (AOAC)	0.7	1.1	1.0	4.0	1.4	1.1	0.6	2.0	0.3	1.3	1.1
P_2O_5 water solubility, % of total	81.6	87.3	68.1	62.7	74.9	85.8	75.4	86.5	61.4	84.6	79.8
Screen analysis (Tyler),[c] %											
+6 mesh	26.0	0.2	0.0	0.0	–	–	–	–	–	–	–
-6 +10 mesh	70.5	76.2	51.5	72.3	–	–	–	–	–	–	–
-10 +16 mesh	3.2	22.5	46.4	22.7	–	–	–	–	–	–	–
-16 mesh	0.3	1.1	2.1	5.0	–	–	–	–	–	–	–
+6 mesh	–	–	–	–	2.3	3.0	0.2	4.1	0.6	7.3	13.9
-6 +10 mesh	–	–	–	–	69.5	87.8	60.8	77.4	47.3	88.0	81.3
-10 +12 mesh	–	–	–	–	15.2	7.4	35.6	12.5	31.7	2.2	2.5
-12 mesh	–	–	–	–	13.0	1.8	3.4	6.0	20.4	2.5	2.3

[a]45.5 kg/hr (100 lb/hr) pilot plant.
[b]0.90 mt/hr (1 st/hr) pilot plant.
[c]Screen size changed for large pilot plant.

granulation. A relatively low value for P_2O_5 water solubility (63%) was obtained.

As shown in table 6, similar grades, including 14-14-14 and 10-20-10, were tested in the larger scale pilot plant. Granulation was quite poor with test conditions for 14-14-14 as shown in tests 1 and 2. In both formulations, there was a tendency toward undergranulation and overgranulation. There was sticking in the downstream equipment. Some difficulty was encountered even with the test 4 in which only urea, single superphosphate, and potassium chloride were used. The material was difficult to handle downstream of the granulator. Operation was somewhat improved in test 5 in which a small quantity of ammonia was added along with some diammonium phosphate. The ammonia neutralized the free acid in the single superphosphate and gave some heat. Even this test was considered inferior to the conventional granulation process as the material was somewhat wet and sticky. For the 10-20-10 grade operation was satisfactory only when about 10 kg of ammonia per mt of product (20 lb/st) was added to the granulator. Otherwise the granulator product remained sticky. This quantity of ammonia decreased the water solubility of the phosphate to about 80%.

From data obtained thus far at TVA, the granulation of compounds based on urea and single superphosphate without ammoniation appears to be difficult. It appears that some specific equipment changes would be required for formulations of this type. The drying and feeding of hot single superphosphate to the granulator may be helpful. The use of a small quantity of ammonia in the granulator eliminates many of the operational problems, but lowers the phosphate water solubility to about 80%. TVA plans to conduct further studies of the process since somewhat better results were obtained in the smaller-scale pilot plant. In the small equipment, the granulator retention time was about 20 minutes versus about 5 minutes in the larger unit. A longer retention time in the larger drum granulator might improve granulation and decrease stickiness of the product.

Pan-Granulated Urea-Ammonium Sulfate

Ammonium sulfate is considered to be an excellent source of nitrogen, especially in regions where rice is grown. In addition, it is a very good sulfur source. Due to a much higher analysis, urea is gaining in popularity for essentially the same cropping conditions. Urea is a somewhat fragile material and improvements in handling properties are needed. It was felt that a compromise material might be developed based on mixtures of urea and ammonium sulfate which would have certain favorable characteristics of each material. The mixed product would have a higher nitrogen content than ammonium sulfate, contain available sulfur, and possibly have better physical properties than straight urea. TVA initiated studies to determine if a suitable and relatively simple process could be developed to produce such a material. The studies were carried out in two basic types of equipment, a pan granulator and an oil prilling tank. These processes and products are described below.

Process—This process was studied in the large granulation pilot plant in which the production rate was about 0.45 mt/hr (0.5 st/hr) in a 96.5-cm (38-in) diameter pan granulator (figure 3). Most of the test work was carried out for production of a 40-0-0-4S material, although some tests were made of a 35-0-0-16S grade. In these tests, a concentrated urea solution (about 99%) was prepared by

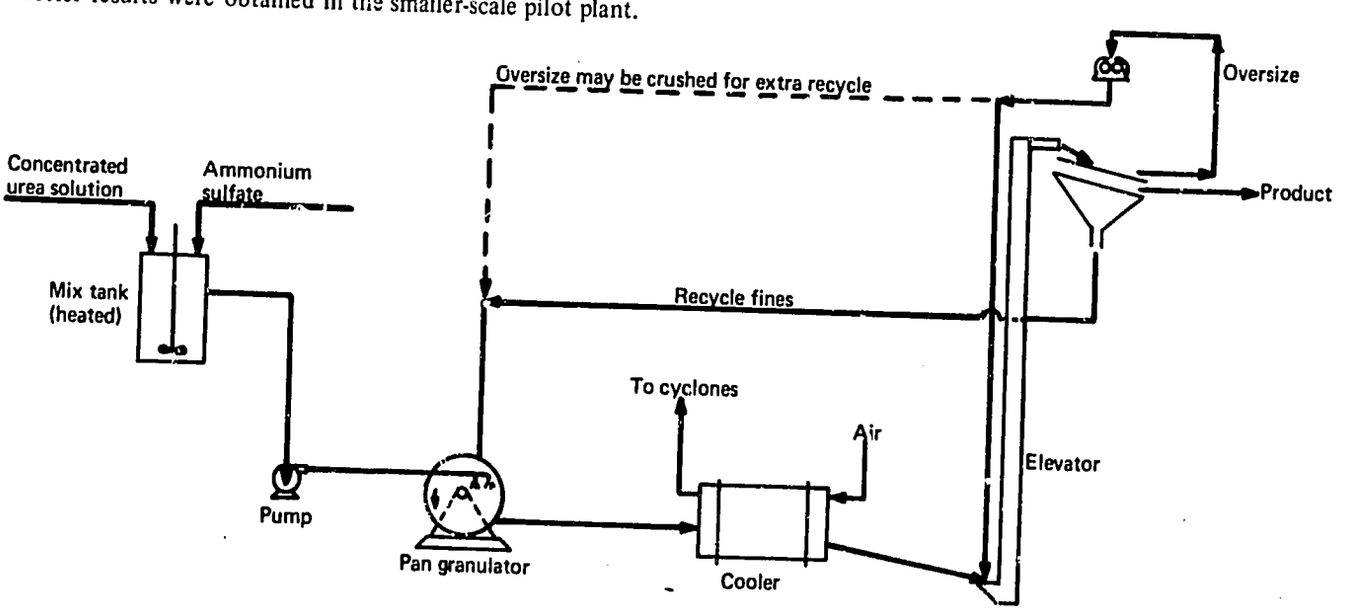

Figure 3. Flow diagram for pan-granulated urea-ammonium sulfate

Table 7. Operating conditions for production of pan-granulated urea-ammonium sulfate (40-0-0-4S nominal grade)

Test no.	1	2
Nominal production rate, mt/hr (st/hr)	0.36(0.4)	0.45(0.5)
Solution preparation		
Feed rate, kg/hr (lb/hr)		
Urea prills	289.5(637)	354.5(780)
Ammonium sulfate crystals	74.1(163)	91.8(202)
Water	4.5(10)	4.5(10)
Solution concentration, % by wt[a]	98.9	99.3
Solution temperature leaving tanks, °C (°F)	152(307)	148(299)
Solution temperature at nozzles, °C (°F)	142(288)	140(284)
Pan granulator		
Diameter, cm (in)	96.5(38)	96.5(38)
Rpm	36	35
Slope, degrees from horizontal	61	61
Temperature of discharge, °C (°F)	99(211)	106(223)
Recycle ratio, kg/kg (lb/lb) product	1.9	1.7
Recycle temperature, °C (°F)	51(124)	54(130)
Cooler exit temperature, °C (°F)	64(148)	68(154)

[a]Solution sprayed by two No. 8 "Whirljet" nozzles, manufactured by the Spraying Systems Company.

melting unconditioned urea prills in steam-heated tanks. This solution was fed by a centrifugal pump to two spray nozzles arranged to deliver solution to the moving bed of recycle. The solid ammonium sulfate is added to the urea prills prior to melting. This method appeared to be superior to feeding the ammonium sulfate directly to the granulator. By use of a concentrated urea solution no dryer is needed. Granules flow from the pan granulator to the cooler and screens. The oversize is crushed and mixed with recycle fines.

Typical Operating Conditions—Table 7 shows some typical operating conditions during production of a 40-0-0-4S urea-ammonium sulfate product in the pan granulator at about 0.45 mt/hr (0.5 st/hr). The solution concentration to the spray nozzles was about 99%. The temperature in the tanks was around 148°-152°C (299°-307°F) and about 140°C (284°F) at the spray nozzles. To obtain a large quantity of on-size product, the recycle ratio was in the range of 1.7 to 2.0 kg/kg (lb/lb) of product. Recycle temperature was about 54°C (130°F); some heat was added to the system to maintain this temperature which should be close to that obtained in a large-scale plant.

At the conditions of operation, part of the ammonium sulfate was actually dissolved. No problem was encountered with settling of crystals in the mixing-melting tank.

Results—As seen in table 8, a high proportion of the granulator product (58-84%) was in the range of minus 5 plus 8 mesh. The granules were rough in appearance compared with pan-granulated urea, but this should not

Table 8. Results of tests for production of pan-granulated urea-ammonium sulfate (40-0-0-4S nominal grade)

Test no.	1	2
Nominal production rate, mt/hr (st/hr)	0.36(0.4)	0.45(0.5)
Screen analysis (Tyler), %		
Granulator discharge		
+5 mesh	2.7	3.1
-5 +8 mesh	83.5	58.1
-8 +10 mesh	12.7	32.3
-10 mesh	1.1	6.5
Onsize product		
+5 mesh	0.0	0.1
-5 +6 mesh	8.2	2.9
-6 +8 mesh	80.6	70.9
-8 +10 mesh	11.2	26.0
-10 mesh	0.0	0.1
Chemical analysis of product, wt %		
Total N	41.6	41.4
Sulfur	4.5	4.6
H_2O (Karl Fischer)	0.3	0.3
Bulk densities, kg/m³ (lb/ft³)		
Pan product	640(40)	672(42)
Cooler product	704(44)	736(46)
Recycle	704(44)	752(47)
Onsize product	688(43)	720(45)

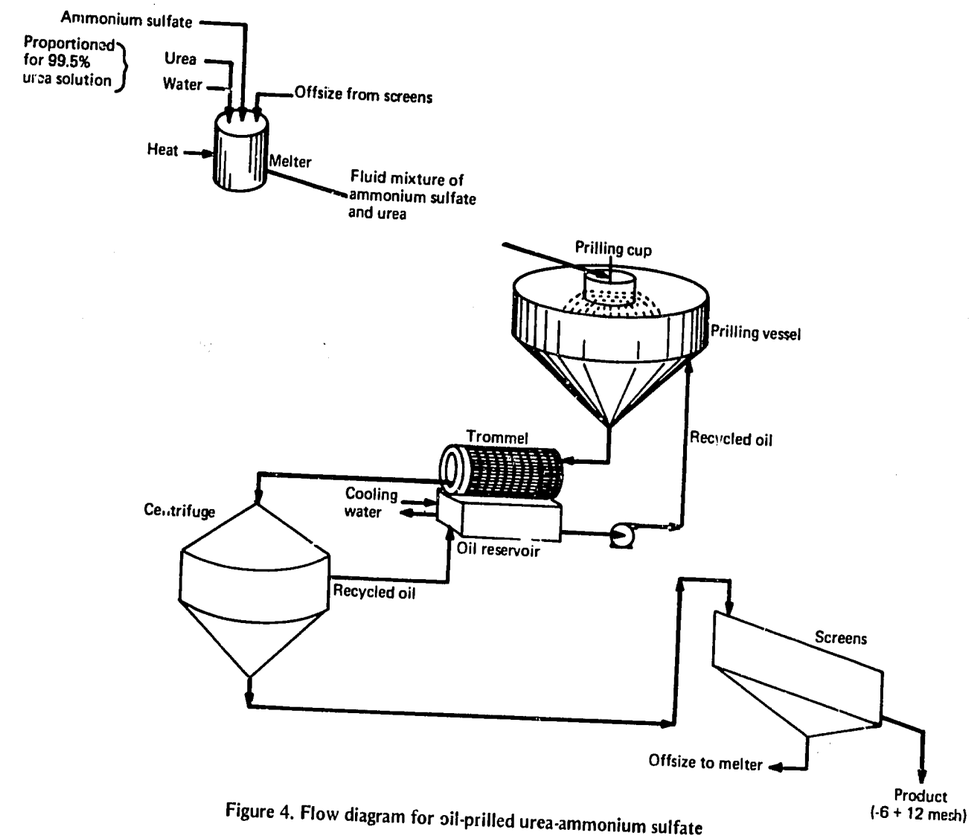

Figure 4. Flow diagram for oil-prilled urea-ammonium sulfate

detract from its use in bulk blending or direct application. Some changes were made in operating conditions and equipment in an attempt to improve surface smoothness, but little success was realized.

The granules had a crushing strength of about 1.8 kg (4 lb) which compared favorably with pan-granulated urea and was of the order of two to three times the crushing strength for air-prilled urea. Moisture content of the product was about 0.3%.

Pan-granulated urea-ammonium sulfate of 40-0-0-4S grade (0.1-0.2% H_2O) would, on the basis of small-scale storage tests, have to be conditioned (preferably with a high-efficiency conditioner such as diatomaceous earth) to remain in satisfactory condition in bulk or bag storage for longer than a month. Precautions should be taken when the material is stored in bulk as moisture would be absorbed somewhat like ammonium nitrate. Protection could be provided in the form of a tightly closed building, plastic pile covers, or dehumidified storage. The critical relative humidity was in the range of 55-60%.

Oil Prilled Urea-Ammonium Sulfate

Oil prilling of some fertilizer materials, such as calcium nitrate, is practiced commercially in some European countries. Tests were made at TVA to demonstrate the technical feasibility of oil prilling urea-ammonium sulfate mixtures. Grades such as 40-0-0-4S (20% ammonium sulfate), 34-0-0-9S (43% ammonium sulfate), and 30-0-0-13S (59% ammonium sulfate) were produced in the equipment described below. The process and products are described briefly in this section. As in the case of air prilling, oil-prilled products are formed as spherical particles having a relatively smooth surface, but some residual oil remains in the product which is not the case in air prilling.

Some limited exploratory tests were made to simulate air prilling of urea-ammonium sulfate. TVA does not have an air-prilling tower, but the simulated tests indicated that air-prilling would be technically feasible.

Process—Figure 4 shows the flow diagram and basic equipment of the pilot plant for the production of

Table 9. Operating conditions for production of oil-prilled urea-ammonium sulfate

Test no.	1	2	3
Nominal grade	40-0-0-4S	34-0-0-9S	30-0-0-13S
Melt-mix tank			
Feed rate, kg/hr (lb/hr)			
Urea	30.9(68)	51.8(114)	21.4(47)
Ammonium sulfate	10.9(24)	39.1(86)	35.9(79)
Temperature, °C (°F)	149(300)	138(280)	138(280)
Retention time, min.	4	3	4
Prilling			
Prill oil	3190[a]	3190[a]	Mineral seal[b]
Prill cup speed, rpm	300	400	400
Inlet temperature to cup, °C (°F)	139(282)	135(275)	132(270)
Prill-tank temperature, °C (°F)	41(105)	38(100)	29(84)
Urea solution concentrate, %	99.8	99.3	99.8

[a]Used in cold-rolling of aluminum, referred to as a roll oil and primarily paraffinic.
[b]Roll oil; somewhat less viscous than 3190 roll oil.

urea-ammonium sulfate by oil prilling. Solid ammonium sulfate (99% -35 mesh) is metered and mixed with 99.5% urea solution in a melt-mix tank. The slurry is fed to a rotating prill cup which has holes allowing the melt to discharge as droplets into a cool-oil medium. The oil quickly cools or quenches the melt in the form of spherical particles. The oil and prill slurry flows to a trommel (wide-mesh rotating screen) where much of the oil is removed. Following the trommel a centrifuge further reduces the oil content of the prills to a low level. Oil from the trommel and centrifuge is collected, cooled, and recycled to the prilling tank. De-oiled prills are screened and offsize material (-12-mesh fines and +6-mesh oversize) is recycled back to the melter. The process was carried out continuously. The pilot plant production was about 68 kg/hr (150 lb/hr) of product. Numerous tests were made with the main objectives of obtaining data for scale-up and to decrease the quantity of residual oil remaining on the prilled product.

Typical Operating Conditions—In order to simulate a concentrated urea solution (99.5% urea), urea prills were melted at about 138° to 149°C (280°-300°F) and mixed with the ammonium sulfate (99% -35 mesh) (table 9). The retention time in the melt-mixer was about 4 minutes. The temperature of the oil in the prill tank was kept in the range of 29° to 41°C (84°-105°F) by cooling. The prill cup 7.6 cm in diameter (3 in) was rotated in the range of 300 to 400 rpm. Several variations of prill cups were tested to determine the effect of hole opening, rpm, and number of holes. The main effect was noted from rpm of the prill cup in the range of variables studied. It was found that the maximum amount of material that could pass through each hole in the prill cup was 0.86 to 1.05 kg/hr/mm² (1.9 to 2.3 lb/hr/mm²) of hole area. Operation of the centrifuge was best in the range 350 to 600 G; prill breakage was excessive when the force was 800 G. Lightweight oils (roll oil 3190 and mineral seal oil) of the type used in cold rolling of aluminum plate were used as prilling medium.

Table 10. Results for oil-prilling of urea-ammonium sulfate

Test no.	1	2	3
Nominal grade	40-0-0-4S	34-0-0-9S	30-0-0-13S
Screen analysis (Tyler), %			
Trommel discharge			
+6 mesh	4	1	7
-6 +10 mesh	71	85	71
-10 +12 mesh	7	11	8
-12 mesh	18	4	14
Oil content, % by wt	37	23	23
Product chemical analysis, %			
Total N	39.0	33.7	30.2
NH_3-N	5.0	8.7	12.9
Total S	6.2	10.0	14.7
Bisulfate S	0	–	0.01
H_2O (Karl Fischer)	0.2	0.3	0.2
Oil	1.6	1.5	1.9
Biuret	1.1	–	0.6
% of prills as ammonium sulfate	26	42	67
Bulk density, kg/m³ (lb/ft³)	800(50)	816(51)	896(56)

Results—Typical results of tests for oil-prilled urea-ammonium sulfate are given in table 10 for three grades of products. Materials taken from the trommel (primary oil separator) were 80% -10 +12 mesh in size and had an oil content of 30% or higher. The product analysis was reasonably close to grade. The moisture content was quite low (0.2-0.4%).

Fresh products had an oily appearance except when conditioned with about 2% of conditioner such as diatomaceous earth or calcined Fuller's earth. After storage up to several days, the oily appearance disappeared without conditioner. The critical relative humidity of 40-0-0-4S and 34-0-0-9S was 55-60% at 30°C (86°F). Bulk density was about 800 kg/m³ (50 lb/ft³) compared with air-prilled urea of 720 kg/m³ (45 lb/ft³). Angle of repose for the materials was 32-37 degrees. The crushing strength of urea-ammonium sulfate containing 9-64% ammonium sulfate was 1.4-1.8 kg (3-4 lbs) as made and after storage, whereas air-prilled urea has a crushing strength of 0.68-0.91 kg (1.5-2.0 lbs).

Bag storage tests were made for oil-prilled urea-ammonium sulfate for 30-0-0-9S and 40-0-0-4S grades for periods up to 9 months. Test results indicated that bulk or bag storage of the oil-prilled products containing 34% nitrogen (0.3-0.4% H_2O) would be satisfactory without use of a conditioning agent. A maximum of 17% light lumps formed on 6-month storage; but, since the lumps were only light, little difficulty should be encountered, especially in bulk handling. Conditioning with 2% of diatomaceous earth or kaolin clay essentially eliminated all lump formation. The bulk piles should be protected against moisture pickup, however, in the same manner as that usually provided for bulk-stored ammonium nitrate. Products of 40% nitrogen content (0.1-0.2% H_2O) would, on the basis of the test results, have to be conditioned (preferably with a high-efficiency conditioner such as diatomaceous earth) to remain in satisfactory condition in bag or bulk storage for longer than a month.

Urea with Improved Physical Properties

The attractiveness of urea as a nitrogen fertilizer could be increased even further if simple and inexpensive methods could be found to significantly improve its physical properties, especially for handling, storage, and transport in humid areas. A urea that is readily compatible with granular triple superphosphate for bulk blending would be helpful. This, of course, would be attractive for industry in the United States as well as in developing countriees. With these objectives, TVA has been conducting tests to evaluate various materials that could be applied to urea after the product has been air-prilled, spheroidized, or pan-granulated. This mainly involves application of a coating to the particle surface in small quantities with evaluation of this coating as a moisture barrier. This is not part of the sulfur-coated urea project in which the objective is to control the dissolution rate of urea in the soil, although thin sulfur coatings may be effective under some conditions.

The Procedure—Numerous materials including oils, waxes, and rosins have been evaluated. The materials are applied, usually in the form of liquids, to the urea particles while tumbling in a rotary drum. The liquids were applied by the most simple and convenient means such as by pouring or spraying. A screening procedure has been used to evaluate the coating as applied in laboratory (small pilot plant) equipment. Coatings with promise have been tested in a larger-scale pilot plant.

The screening or evaluation to determine the effectiveness of particular coatings was done by exposing the coated products to vigorously agitated air at 90% relative humidity and 32°C (90°F) and measuring the moisture absorbed per square centimeter (cm²) of exposed surface. Also the depth of moisture penetration into a cylinder of fertilizer was determined. This procedure and equipment for these tests are described in TVA special report S-444. The moisture absorbed and moisture penetration were determined after 4 and 24 hours, along with visual observations. Other tests were conducted in two controlled temperature humidity rooms. In one room the relative humidity was kept at 90% at 32°C (90°F) constantly, while in the other an 8-hour period was at 90% relative humidity at 32°C (90°F) and a 16-hour period at 32°C (90°F), 60% relative humidity. Larger samples were exposed in these rooms. Approximately 200 samples have been prepared for evaluation. Some of the more promising materials are discussed below. The coated product was assumed to show promise if it absorbed no more than 25 mg H_2O/cm^2 in 4 hours and no more than 125 mg H_2O/cm^2 in 24 hours in the laboratory tests at 90% relative humidity and 32°C (90°F).

Results—Table 11 gives some typical results of exposure tests with uncoated and coated spheroidized urea in the laboratory studies. Several types of coatings were promising as materials to retard moisture absorption at 32°C (90°F) and 90% relative humidity. Normal sulfur-coated urea (20% coating) was outstanding and only absorbed 3 and 17 mg/cm² in 4 and 24 hours, respectively. Other materials such as Bunker C fuel oil, mixtures of paraffin and oil, and mixtures of petrolatum, rosin, and paraffin were effective in the laboratory tests. One of the least expensive materials, Bunker C fuel oil, gave results that were about the same as more expensive materials, but when conditioner was used on top of the oil, moisture resistance was decreased somewhat. At the 2% coating level for oil, a conditioner would be needed to prevent tackiness.

Larger quantities of materials were made in the pilot plant so that exposure tests could be made with larger samples. Some typical results are shown in table 12. Samples were exposed for 7 and 21 days under two conditions of controlled temperature and humidity. At 32°C (90°F) and 90% relative humidity, unconditioned urea absorbed water at the rate of 745 and 2683 mg/cm² in 7 and 21 days, respectively; the total depth of 34.3 cm (13.5 in) was composed of a hard, wet cake. Three types of materials described in the table reduced the absorption by

Table 11. Resistance of some coated urea products to a humid atmosphere—laboratory evaluation[a]

Test no.	Coating material	Application temp., °C (°F)	Amount applied, wt %	Method of application	Product N, %	Hygroscopicity: Moisture absorption mg/cm² Hours 4	24	Moisture penetration, cm Hours 4	24	Observed appearance of wetted material after indicated exposure Hours 4	24
1.	Spheroidized urea, uncoated	–(–)	–	–	46.0	53	293	1.5	B	Wet	Mud
2.	Spheroidized urea, uncoated, fire polished	–(–)	–	–	46.0	77	383	2.0	B	Wet	Mud
3.	Normal sulfur-coated urea	–(–)	20.0	Spraying	37.0	3	17	0.0	0.0	Ok	Ok
4.	Oil-prilled urea in 10% Fisher paraffin + 90% 2110 oil	60(140)	2.4	Prilling	45.4	9	38	1.0	3.5	Damp	Damp
5.	Spheroidized urea coated with a mixture of 10% soybean oil, 10% Fisher paraffin, and 80% 2110 oil	60(140)	2.0	Spraying	45.2	11	49	0.4	1.0	Ok	Ok
6.	Spheroidized urea coated with a mixture of 1:3:1 Texaco 1231, Tenneco Doubl. and Texaco 10S	93(200)	2.5	Pouring	45.0	12	44	0.5	1.3	Damp	Damp
7.	Spheroidized urea coated with a mixture of 10% linseed oil, 10% Fisher paraffin, and 80% 2110 oil	60(140)	2.0	Spraying	45.5	16	68	0.8	3.2	Ok	Wet
8.	Spheroidized urea coated with Bunker C fuel oil	60(140)	2.0	Pouring	45.5	19	89	1.4	4.0	Wet	Mud
9.	Spheroidized urea coated with a mixture 90% petrolatum-rosin-paraffin (PRP) + 10% soybean oil	82(180)	2.0	Pouring	45.5	14	52	0.2	2.0	Ok	Wet
10.	Spheroidized urea coated with a mixture 10% Shellmax 800, 10% gilsonite, and 80% 2110 oil	38(100)	2.0	Spraying	45.5	18	78	0.8	2.5	Damp	Damp

[a]Samples exposed in cylindrical glass cups 6.8-cm-dia. x 5.1-in-deep (2.68-in-dia. x 2-in-deep) in controlled temperature humidity cabinet; spheroidized urea of size -6 + 10 mesh.

Table 12. Resistance of some coated urea products to a humid atmosphere—pilot plant evaluation[a]

Test no.	Type of coating	Exposure at 32°C (90°F), 90% RH: Moisture absorption per unit of exposed surface, mg/cm² 7 days	21 days	Depth and condition of layer, cm After 7 days	Exposure conditions cycled daily: 8 hr at 32° C (90°F), 90% RH, then 16 hr at 32° C (90°F) and 60% RH: Moisture absorption per unit of exposed surface, mg/cm² 7 days	21 days	Depth and condition of layer, cm After 7 days
1.	Unconditioned prills	745	2683	34.3(13.5-in) hard, wet	79	282	34.3(13.5-in) hard
2.	Conditioned prills (2% kaolin)	654	2388	34.3(13.5-in) hard, wet	42	148	2.0(0.8-in) moderate
3.	Conditioned granular (1% diatomaceous earth)	756	2604	20.3(8-in) hard, wet	59	209	2.5(1-in) hard
4.	Granular with a 0.9% coating of 10% soybean oil + 10% paraffin + 80% 2110 oil	446	1468	12.7(5-in) moderate	–	47	2.5(1-in) light
5.	Granular with a 2.4% coating of 45% Shellmax 800 wax + 55% 2110 oil with a 1.5% coating of diatomaceous earth conditioner	353	1207	20.3(8-in) light	27	36	1.3(0.5-in) light
6.	Granular with a 3.9% coating of a 1:3:1 mixture of petrolatum, rosin, and paraffin	338	1039	12.7(5-in) loose	19	31	1.3(0.5-in) lihgt

[a]Samples exposed in level-full, open-top cans 31.8-cm-dia x 34.3-cm-deep (12.5-in-dia x 13.5-in-deep) in controlled temperature humidity rooms.

about 50%. A coating of wax and oil topped with conditioner, as well as a coating of a 1:3:1 mixture of petrolatum:rosin:paraffin, was considered effective, but rather expensive. These coatings were calculated to cost $2.73 and $6.38/ton, respectively.

Work is continuing on improvement of physical properties of urea with the objective of finding a coating that will be effective in dosages small enough to give 45% nitrogen product, that will limit absorption to a degree equal to the above coatings, but will cost only in the order of $1.50-$2.00/ton for the coating material, not including the cost of application. One material that appears especially promising is a 1% coating of a mixture of wax (62°C; 143°F melting point) and Bunker C fuel oil which would cost no more than $0.50/ton. One possible disadvantage of this material is that a discoloration of the urea is noted when this material is used.

Applications—As pointed out previously, an improved urea would have good potential for developed as well as developing countries. Perhaps the largest need is for a urea product that could be blended with unammoniated granular triple superphosphate. However, almost any treatment, coating, conditioner, or whatever, will increase product cost. No one seems prepared to say what increased cost can be tolerated, although some have suggested that a product containing as little as 40% nitrogen and costing 10% more per unit of nitrogen might be acceptable. Sulfur-coated urea prepared to control the dissolution rate of nitrogen has been shown to be compatible with granular triple superphosphate as well as resistant to extreme conditions of humidity and temperature, but the cost of this type coating could not be tolerated solely for improvement of physical properties.

Granulated Fine-Ground Rock Phosphate

Some very limited tests were made to determine the feasibility of granulating fine-ground phosphate rocks by partial acidulation. It was felt that some water-soluble phosphate in the product would be helpful for early stages of plant growth. The acidulated portion would provide this. Phosphate in unreacted rock would become available for crop use over the long term.

Tests were conducted in laboratory bench-scale equipment and in the pilot plant. Only data from the pilot-plant pan granulator are included in this report. To supply liquid phase and binder for granulation and to obtain some water-soluble phosphate in the product, sulfuric acid was sprayed onto a moving bed of fine-ground rock in the pan granulator. The quantity of sulfuric acid was limited to about 25% of that required for acidulation to make single superphosphate. For convenience, the sulfuric acid and water were sprayed using separate nozzles to give acid concentrations after mixing in the range of 29 to 33%. Two rocks were used: Uncalcined North Carolina (29.9% P_2O_5; 89% -200-mesh) and uncalcined Central Florida (32.7% P_2O_5; 68% -200-mesh). The spray water was preheated to 82°C (180°F) and a direct flame was used on the rolling bed of granules. Product was dried at 82°C (180°F) and cooled to 27°C (80°F) before screening. Product was screened on 5- and 14-mesh screens. Typical operating conditions for production of partially acidulated ground rock phosphate are shown in table 13.

Some degradation of granules occurred in the dryer and cooler, which decreased the quantity of onsize material (-5 +20 mesh). Apparently the granules were relatively weak and not considered of comparable quality with usual mixed fertilizer granules; the crushing strength was about 0.68 to 0.91 kg (1.5-2.0 lb). The product made from North Carolina rock contained 26% total P_2O_5 of which 10.6% was soluble in neutral ammonium citrate and 5.3% in water. With Central Florida rock the total P_2O_5 was 28.1% of which 10.4% was soluble in neutral ammonium citrate and 6.1% soluble in water.

Materials for Agronomic Tests

Some of the experimental materials made in the various pilot plant tests at TVA are kept on hand for agronomic evaluation. Small quantities of these materials can be made available to research workers who are interested in evaluating their agronomic characteristics. Requests for samples should be addressed to D. L. McCune, TVA–NFDC, Muscle

Table 13. Operating conditions for granulation of fine-ground rock phosphate by partial acidulation[a]

Test no.	1	2
Rock source	North Carolina	Central Florida
Rock analysis, %		
CaO	48.6	47.5
P_2O_5	29.9	32.7
F	3.5	3.6
CO_2	5.4	3.3
Al_2O_3	0.46	1.20
Fe_2O_3	0.68	1.45
SiO_2	1.6	5.2
Pan granulator conditions		
Slope of pan, degrees from horizontal	55	61
Rpm	24	27
Feed rate, kg/hr (lb/hr)		
Rock	304.5(670)	300(660)
Acid (93% H_2SO_4)	45.5(100)	45.5(100)
Water	99.5(219)	84.5(186)
Acid concentration (acid + water), % H_2SO_4	29	33
Temperature, °C (°F)	(142)	(151)
Dryer temperature, °C (°F)	76(169)	84(184)
Cooler temperature, °C (°F)	27(80)	27(80)

[a]96.5-cm diameter (38-in) pan granulator.

Shoals, Alabama. The researcher is asked to provide information about how the samples are to be used and to share his research findings with TVA. On this basis the materials are free, but the research worker or his organization is expected to pay the freight cost involved.

A partial listing of other typical experimental materials not described in this report but which are available for agronomic testing is given in tables 14 and 15. Further information about these or other materials may be obtained by writing to the above address.

Table 14. Typical experimental materials available for agronomic testing

Plant food ratio	Grade	Micronutrient added
Ammonium orthophosphates		
1:4:0	13.5-56.5-0	Zn, Cu, Mn, Mo, B
Ammonium polyphosphates		
1:4:0	13.5-52.5-0	Zn, B, Cu, Mn, Mo
3:4:0	20.5-27.4-0	Mn
Mixed fertilizers		
1:4:2	3.5-15.0-7.0	Mn, Zn
0:1:0	0-38.5-0	S, Na
Custom blends		
0:1:1	0-25-23	None
2:1:0	28.6-14.3-0	None
Urea-ammonium polyphosphates		
1:2:0	22.0-46.0-0	None
1:1:0	30.0-30.0-0	None
2:1:0	36.0-18.0-0	None
Urea-amoniated triple superphosphates		
1:2.5:0	14.0-35.0-0	None
4:6:0	20.0-27.0-0	None
Sulfur-coated materials[a]		
Urea	Various[a]	None
Ammonium phosphate nitrate	20.7-030S or 19-6-030S	None
Ammonium polyphosphate	13-55-10S or 13-51-015S	None
Nitric phosphates		
1:1:0	19.6-20.0-0	None
1:2:0	15.4-28.2-0	None
2:1:0	26.1-13.2-0	None
Urea-ammonium phosphate		
3:1:0	38.5-12.9-0	None
1:1:0	29.9-28.2-0	None
2:1:0	35.0-18.3-0	None
1:2:0	22-42-0	None

[a]Products are available having a range of coating weights and dissolution rates.

Table 15. Ground rock phosphate for agronomic evaluation

Source	Chemical analysis, %			Screen analysis (Tyler), %					
	P_2O_5	Citrate-Soluble P_2O_5[a]	CaO	+65	-65 +100	-100 +150	-150 +200	-200 +325	-325
Idaho	32.3	3.7	46.8	0.0	0.3	0.5	3.5	15.5	80.2
Central Florida	32.7	5.3	47.5	0.0	4.2	7.8	20.2	16.8	51.0
North Carolina	29.9	7.8	48.6	0.0	2.0	3.2	6.0	9.8	79.0
North Florida	32.4	6.6	48.3	15.8	8.7	6.8	9.5	10.5	48.7
Tennessee	30.1	4.5	42.3	12.0	10.5	9.0	12.5	12.5	43.5
Missouri	34.7	0.5	50.1	0.0	2.0	10.5	30.0	33.5	24.0
India	40.1	2.1	54.2	5.2	5.5	4.7	5.3	5.5	73.8
Morocco	33.3	–	51.0	19.7	18.5	19.7	7.1	5.7	19.6

[a]Neutral ammonium citrate method.

SUMMARY

The technical feasibility for production of some experimental fertilizer materials which have potential application in developing countries has been demonstrated. Materials such as granulated urea-phosphate rock and urea-ammonium sulfate probably could be produced in developing countries, although process details have not been worked out for large-scale production. No significant problems were encountered in pilot-plant tests which would prevent further scale-up. The production of urea-single superphosphate-based granules without ammoniation is a difficult process to control, and additional pilot-plant tests are needed to overcome operational problems. Some equipment modifications might improve operation. Tests thus far are promising for development of a coating material which will improve the physical characteristics of urea used under tropical conditions.

TVA plans to continue work on some of these materials if funds are available.

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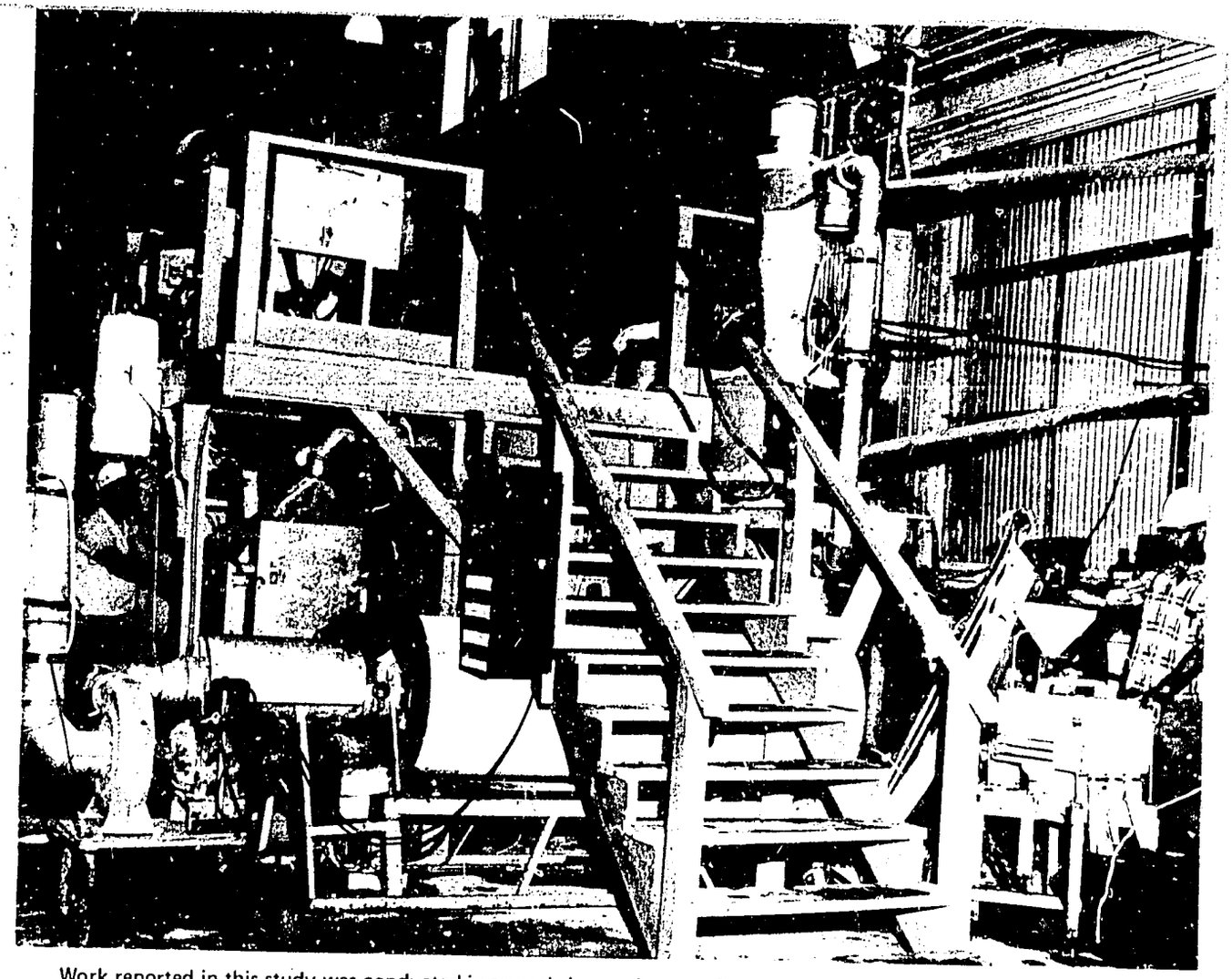
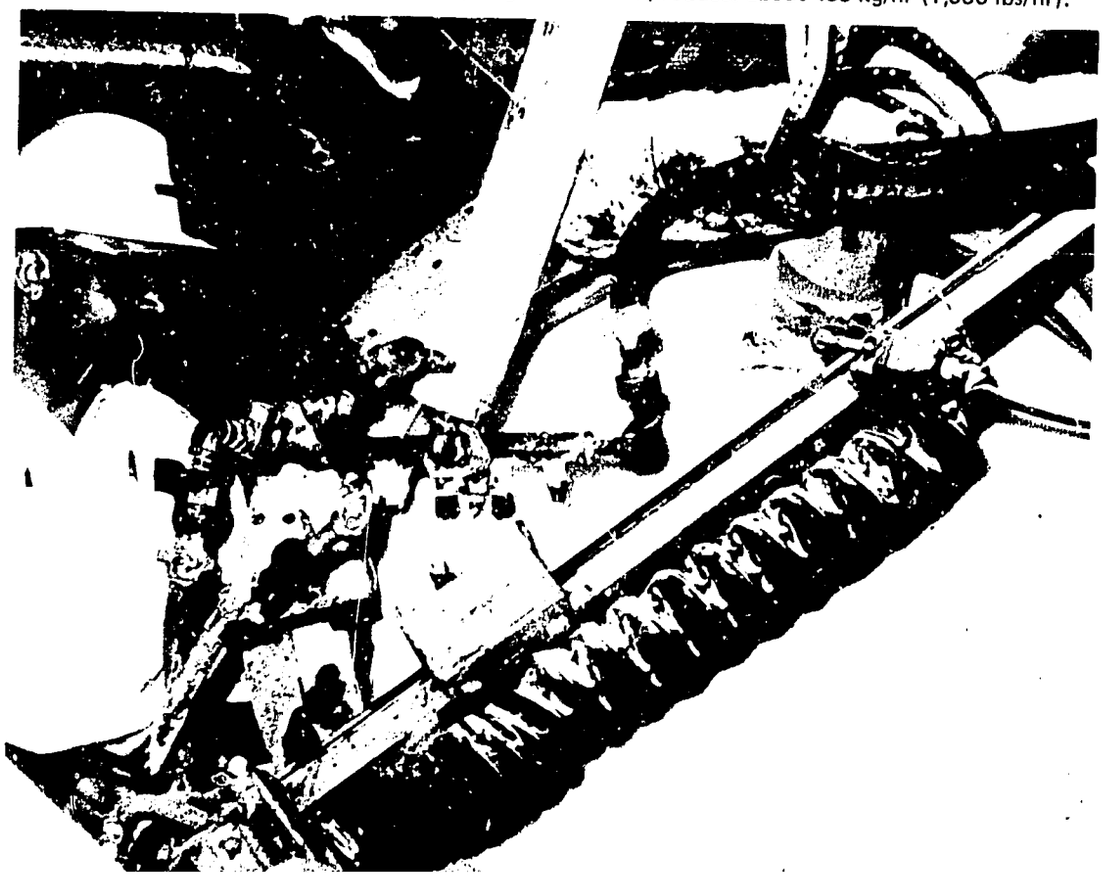


Work reported in this study was conducted in several sizes and types of laboratory equipment and pilot plants, two of which are shown in these photographs. Above is the intermediate size drum granulation pilot plant. It produces about 45 kg/hr (100 lbs/hr). Below is a 38-inch-diameter pan granulator. It produces about 450 kg/hr (1,000 lbs/hr).



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