

Effectiveness of the water-insoluble component of triple superphosphate for yield and phosphorus uptake by plants

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SUMMARY

The water-insoluble phosphorus (P) component ('residue source') was separated from four commercial triple superphosphate (TSP) products and its agronomic effectiveness evaluated. Two of the TSP products were sourced from the USA and two from the UK. Effectiveness was measured using a pot trial technique with ryegrass as the test crop. The residue sources were evaluated on 13 soils varying in key properties: readily plant-available P, texture, pH and organic matter content. Four of the soils were from Germany, one from Spain, five from the UK and three from France. Grass dry-matter yield and P offtake were measured at every cut on every soil. For both of these variables, significant responses to applied P were obtained on all soils. Mono-calcium phosphate (MCP) was used as the comparative P source and effectiveness of the residue sources was estimated relative to that of MCP. Using dry-matter data, the residue sources were 44–87% as effective as MCP; P offtake data provided corresponding estimates of 35–79%. Both methods of estimation indicated the same ranking of residue sources in terms of relative effectiveness, those derived from USA products being more effective than those derived from UK products. The ranking was the same for all soils. The effectiveness of the residue sources relative to that of MCP appeared little affected by measured soil properties: texture, pH, available P or organic carbon content. The results indicate there is little difference in agronomic effectiveness of TSP products with water solubility greater than 85%.

INTRODUCTION

In 1842, J. B. Lawes of Rothamsted took out a patent for the manufacture of single superphosphate and was producing it commercially in a factory in London by 1843 (Johnston 1994*a*). Much of the phosphorus (P) in this product was water-soluble mono-calcium phosphate (MCP) that was shown to be highly effective in increasing yields of farm crops on most soils. Efficacy and water solubility appeared to be associated. Later, Lawes indicated that superphosphate should be sold with a guarantee of its percentage water solubility. This would be a form of warranty for farmers who, at that time, had little knowledge of fertilizers. Low water solubility implied either adulteration of a well-made product with an inert,

valueless material or a product made with too little sulphuric acid so that the phosphate rock was not all converted to MCP. In the UK, the need to declare the percentage water solubility of superphosphate was enshrined in the first Fertilisers and Feedingstuffs Act in 1893 and remained so in subsequent Acts but without mention of a minimum value. Similar requirements were common in other European countries. Subsequently, the concept of 'harmonization' in European Union countries required manufacturers wishing to sell a product as an EC-Type fertilizer to comply with certain technical standards (EC 1976). The criteria for superphosphates, including triple superphosphate (TSP), included the requirement that the water-soluble P₂O₅ must be at least 93% of the P₂O₅ soluble in neutral ammonium citrate (NAC). This requirement had the effect of not allowing TSPs made from some phosphate rocks to be labelled as EC-Type fertilizers.

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Much earlier than 1976, research on a range of alternative P sources with varying water solubility for use in agriculture had questioned the need for a large proportion of the P to be water-soluble. For work in the USA, see Terman *et al.* (1956), De Ment & Seatz (1956), Lawton *et al.* (1956) and Webb & Pesek (1958). In Europe, Cooke (1956) reviewed experiments done in 13 European countries on P fertilizers where production economized in the use of sulphuric acid. These fertilizers included (i) dicalcium phosphate dihydrate which, though insoluble in water, was found to be approximately as effective as superphosphate; (ii) lightly ammoniated superphosphates which were fully equivalent to superphosphate although only 70% of the P was water-soluble; (iii) nitrophosphates, then with little water-soluble P, which were often not as effective as superphosphate. Later research showed that the effectiveness of nitrophosphates depended on the method of manufacture. Two nitrophosphates with only 59 and 73% water solubility produced by a Norsk Hydro process (Storen 1992), when compared with a mixture of TSP and monoammonium phosphate having 95% water solubility, gave the same yields of spring barley and 97% of the yield of potatoes (Johnston 1994*b*).

Such results question the need for a minimum of 93% water solubility in superphosphates as required by the EU legislation and easily achieved using rock phosphate from North Africa but not using rock from many other sources. The variability in the water solubility of P in TSP arises from components of the rock, mainly iron and aluminium, reacting with P during the manufacturing process to produce water-insoluble P compounds. Although such compounds can be removed there is both an energy cost and an environmental cost in disposing of them. However, where these compounds exist in TSP there is no problem in adding the small amounts to agricultural soils, many of which contain large amounts of iron and aluminium phosphates that are not known to have any detrimental effect on plants.

There would be less concern about the somewhat lower percentage water solubility in some TSP products if it could be shown that the water-insoluble P component had some agronomic value. It was decided, therefore, to assess the plant availability of the P in the water-insoluble component of TSP products with different levels of water solubility for crops grown on a range of European soils where traditionally TSPs with high water solubility have been used.

METHODS

Sources of treatment materials

Two 10 kg samples of TSP products were obtained from retail sources in the USA (US-TSP) and two

Table 1. *Total and soluble phosphorus (expressed as P₂O₅) in the four TSP products and in the residue sources*

Source	USA	USA	UK	UK
Triple superphosphate analysis				
% total P ₂ O ₅	47.6	48.2	47.0	45.7
% water soluble P ₂ O ₅	40.0	41.2	42.5	43.5
% neutral ammonium citrate soluble P ₂ O ₅	44.2	44.5	44.0	44.9
Water soluble as % total P ₂ O ₅	84.0	85.5	90.4	95.2
Water soluble as % citrate soluble P ₂ O ₅	90.5	92.6	96.6	96.9
Water insoluble component analysis				
Code	US-1	US-2	UK-1	UK-2
% total P ₂ O ₅	31.2	33.4	29.1	14.0
% water soluble P ₂ O ₅	0.89	1.65	0.57	0.50

from the UK (UK-TSP). The two products from the UK were labelled 'EC-Type Fertilizer'. On receipt, subsamples were taken for the determination of total P₂O₅, and P₂O₅ soluble in water and in neutral ammonium citrate (Anon. 1996).

Preparation of treatment materials

The water-insoluble components tested were intended to approximate as closely as possible to that P fraction which is not measured by the standard test for water-soluble P in triple superphosphate (Anon. 1996) and were prepared as follows. A sample of each TSP product was sieved to <0.5 mm using minimal grinding in a mortar and pestle. Then 100 g of the sieved TSP were transferred to a 10 litre plastic wide-neck carboy to which was added 10 litres of demineralized water (20–25 °C). The carboy was rolled to and fro on the bench for 30 min then stood upright before being left overnight at an angle to allow the solids to settle towards the bottom corner. Using a pipette, 100 ml of clear liquid were recovered from mid-carboy, transferred to a polythene bottle, labelled and stored in a fridge. The majority of the liquid was then drawn off from the carboy using a filter pump, taking care not to remove any solid. The remaining contents of the carboy were poured into a buchner funnel fitted with a glass fibre filter paper and the solids were recovered under suction. The carboy was rinsed once with a minimum of demineralized water to recover as much of the solids as possible. The water-insoluble residue was dried overnight at 30 °C,

Table 2. Properties of the 13 soils used in the experiment

Soil number	Country of origin	Available P mg/l and status	pH	CaCO ₃ %	Organic carbon %	Texture	Available K mg/l and status	Available Mg mg/l and status
1	Germany	33 medium	5.8	0.2	1.3	Loamy sand	86 low	94 medium
2	Germany	81 high	7.5	1.3	2.5	Sandy clay loam	203 medium	124 high
3	Germany	54 high	6.0	0.3	1.4	Sandy loam	67 low	96 medium
4	Germany	41 medium	7.3	0.2	1.0	Sandy loam	118 low	178 very high
5	Spain	31 medium	8.1	46.2	0.6	Clay loam	477 very high	134 high
6	UK	17 very low	7.4	1.6	1.1	Sandy clay loam	151 medium	58 medium
7	UK	8 very low	4.9	0	0.7	Clay loam	138 medium	52 medium
8	UK	11 very low	5.3	0	0.7	Silty clay loam	111 low	49 low
9	UK	15 very low	7.4	0.8	0.7	Silty clay loam	109 low	29 low
10	UK	22 low	6.6	0.3	1.9	Clay	159 medium	75 medium
11	France	43 medium	5.8	0	1.6	Sandy silt loam	304 high	169 high
12	France	41 medium	7.7	1.4	0.3	Clay loam	461 very high	103 High
13	France	59 high	8.1	12.0	0.3	Loamy sand	210 medium	63 medium

then removed from the glass fibre filter paper and sieved to <0.5 mm by gentle pressure with a rubber bung. This procedure was repeated until sufficient bulked material had been produced for the pot trials, P analyses and an archive sample of the residue. Total P content was measured on the four bulked residues as follows: 2.5 g were weighed to the nearest 0.001 g and placed in a dry Kjeldahl flask. To this was added 15 ml demineralized water and the flask contents swirled to suspend the solid before adding 20 ml nitric acid and 30 ml sulphuric acid. When the initial violent reaction had ceased, the contents of the flask were brought slowly to boiling point and boiled for 30 min. The flask was allowed to cool and then about 150 ml demineralized water were added with mixing, and boiled for 15 min. The flask was cooled completely and the liquid transferred quantitatively to a 500 ml graduated flask. The contents were made up to volume, mixed and filtered through a fluted filter, discarding the first portion of the filtrate before determining the P in the filtrate using a Technicon auto-analyser (Technicon 1973). Standard solutions were prepared with the same reagent concentrations and all standard and sample solutions were diluted to fall within the concentration range that the auto-analyser could accommodate. Water-soluble P contents of the residues were also measured. For this, 1 g of residue sample was weighed to the nearest 0.001 g, transferred to a shaker bottle to which was added 100 ml demineralized water (temperature range 20–25 °C). The bottle was stoppered, shaken end-over-end for 30 min and filtered before determining P in the filtrate using an auto-analyser. The residue sources derived from US-TSP were labelled US-1 and US-2 and those derived from UK-TSP were labelled UK-1 and UK-2. Total, water-soluble and neutral ammonium citrate-soluble P₂O₅ contents in the TSP products measured by standard UK methods

(Anon. 1996) were in the ranges 45.7–48.2%, 40.0–43.5% and 44.0–44.9% respectively (Table 1).

Soils and treatment fertilizer incorporation

Thirteen soils were obtained for the trials, four from Germany, one from Spain, five from the UK and three from France. All were typical agricultural soils but varied in texture, pH and organic carbon content (Table 2). It was intended that all soils should be low in available P but those from Germany and France had a medium to high P status. Available P was determined by resin extraction (Hislop & Cooke 1968) and available K and Mg by extraction with ammonium nitrate (MAFF 1986). The sand (0.06–2.0 mm), silt (0.002–0.06 mm) and clay (<0.002 mm) contents were measured by a hydrometer method and were converted to a soil texture classification using the UK system (MAFF 1985). Soil pH was measured after 30 min equilibration of a soil:water (1:2.5) suspension (MAFF 1986).

Every soil was sieved to <6 mm and thoroughly mixed before starting the glasshouse experiment. For a given soil, the same weighed amount was placed in five pots. Pots were of 1 litre capacity and held around 1 kg of soil, the exact amount required to fill the pot to within 1 cm of the rim varying slightly depending on the bulk density of each soil. Treatment materials were mixed with the soil for each pot before it was placed in the pot. MCP was included at four rates of application (0.10, 0.15, 0.20 and 0.25 g P₂O₅/pot); the residue materials were applied at 0.20 g P₂O₅/pot. An untreated control was included. A starter amount of 0.05 g N as ammonium nitrate was incorporated with the soil in every pot. A uniform application of 0.01 g Mg was mixed with the soil as magnesium sulphate in soils 6, 7, 8 and 9 where the available Mg concentration was less than 60 mg/l. In every pot, 0.3 g

Table 3. Total dry-matter yield of the grass (g/pot)

Material and amount	Soil												
	1	2	3	4	5	6	7	8	9	10	11	12	13
Control	18.50	26.92	25.13	21.86	19.07	8.90	8.91	9.38	7.93	11.48	13.63	7.21	6.91
MCP 0.10	22.13	27.32	26.07	24.95	23.45	20.07	20.55	18.60	17.64	19.01	14.13	7.23	7.80
MCP 0.15	22.45	28.12	26.41	25.91	24.27	23.18	22.54	21.97	18.49	21.91	14.81	7.84	7.24
MCP 0.20	24.13	28.13	27.25	26.97	24.69	24.83	23.31	24.09	21.91	22.20	14.55	8.15	8.07
MCP 0.25	23.89	29.22	27.48	27.34	25.65	26.51	25.58	25.04	22.16	23.82	15.21	7.51	7.78
US-1 0.20	22.83	28.77	27.02	26.27	23.20	18.78	22.11	21.73	16.83	20.32	13.96	7.45	6.99
US-2 0.20	24.15	28.46	27.43	26.61	25.22	23.46	22.93	24.00	19.30	22.00	14.04	7.68	7.13
UK-1 0.20	22.85	27.57	26.45	25.07	21.07	13.59	21.24	21.25	13.57	16.55	13.94	7.48	6.55
UK-2 0.20	23.50	28.28	27.37	25.63	22.27	17.38	22.43	21.82	15.89	20.52	13.99	6.96	7.17
Mean	22.71	28.09	26.74	25.62	23.21	19.63	21.07	20.99	17.19	19.98	14.25	7.50	7.29
s.e./pot	0.73	0.88	0.63	0.65	0.73	1.24	0.76	0.89	0.77	0.96	0.59	0.46	0.52
D.F.	32	32	32	32	32	32	32	32	32	32	24	24	24

Table 4. Total P offtake in the harvested grass (mg/pot)

Material and amount	Soil												
	1	2	3	4	5	6	7	8	9	10	11	12	13
Control	34.60	62.93	60.54	40.97	31.83	13.18	12.39	13.01	11.51	20.07	41.92	34.97	26.54
MCP 0.10	52.82	77.56	74.80	60.21	52.11	32.06	33.97	33.08	29.30	38.13	51.31	38.56	32.80
MCP 0.15	59.90	87.49	81.71	70.11	61.58	41.41	42.55	42.77	33.72	46.99	54.68	40.55	34.19
MCP 0.20	70.91	94.24	87.28	84.03	68.34	48.26	46.96	52.32	42.64	53.35	57.90	44.00	36.74
MCP 0.25	77.74	103.81	92.62	89.93	72.94	55.08	58.40	60.30	46.63	58.81	62.37	42.48	38.63
US-1 0.20	56.18	77.75	75.59	56.51	46.72	29.50	38.75	36.42	26.95	40.82	46.89	36.27	25.79
US-2 0.20	66.88	86.63	83.81	70.39	61.42	41.18	45.63	47.99	33.68	48.70	53.99	38.55	32.21
UK-1 0.20	52.24	68.58	70.36	50.55	39.79	20.15	35.85	34.67	21.49	33.97	46.59	34.61	26.43
UK-2 0.20	57.35	75.03	73.99	57.04	45.19	25.80	40.23	37.32	24.22	40.09	48.45	34.18	29.78
Mean	38.74	81.56	77.86	64.42	53.32	34.07	39.41	39.76	30.02	42.55	51.57	38.24	31.46
s.e./pot	2.58	3.57	2.97	2.78	2.75	2.90	2.32	3.18	1.77	1.94	2.42	2.15	1.85
D.F.	32	32	32	32	32	32	32	32	32	32	24	24	24

(approximately 150 seeds) of ryegrass (cv. Port Stewart) was sown.

Experimental design

The nine treatments to be applied to every soil comprised MCP applied at 0.10, 0.15, 0.20 and 0.25 g P₂O₅/pot, the four residue sources applied at 0.20 g P₂O₅/pot and a control that received no P₂O₅. Separate pot experiments were established for individual soils, with the nine treatments replicated five times (soils 1–10) or four times (soils 11–13). Trial design was a randomized block and data were statistically treated by analysis of variance.

Management of pots and grass sampling

Pots were maintained in a net-sided polytunnel. All watering was with de-mineralized water. After every harvest of the grass, 0.2 g N as ammonium nitrate was

Table 5. Relative effectiveness (% RE) averaged over the 13 soils

Residue source	% RE derived from	
	Yield	P offtake
US-1	63	42
US-2	82	74
Mean	72	58
UK-1	46	28
UK-2	59	41
Mean	52	35

applied to every pot and after harvest numbers 1, 3, 5 and 7, 0.2 g K₂O as potassium sulphate was applied to every pot. These two fertilizers were applied in solution to the surface of the soil. The applications of N, K₂O and S were intended to prevent any potential

Table 6. Effect of soil available P on relative effectiveness (% RE)

Residue source	% RE derived from							
	Yield				P offtake			
	Soil P category							
	Low	Medium	High	Mean	Low	Medium	High	Mean*
US-1	59	60	75		54	35	34	
US-2	78	84	86		81	70	70	
Mean	68	72	80	72	68	52	52	58
UK-1	44	41	59		40	22	17	
UK-2	57	45	87		51	34	38	
Mean	50	43	73	52	46	28	28	35
Overall mean	59	58	76		57	40	40	

* The overall mean in this column is not the arithmetic mean of the low, medium and high columns because there was a different number of soils in each group.

Table 7. Effect of soil pH on relative effectiveness (% RE)

Residue source	% RE derived from					
	Yield			P offtake		
	Soil pH					
	<6.5	6.5-7.5	>7.5	<6.5	6.5-7.5	>7.5
US-1	75	72	27	53	46	16
US-2	91	87	60	85	75	55
Mean	83	80	44	69	60	36
UK-1	59	50	18	44	26	4
UK-2	76	67	18	55	41	14
Mean	68	58	18	50	34	9
Overall mean	76	69	31	59	47	22

deficiencies in these nutrients affecting the growth of the grass. The grass was sprayed with insecticide whenever aphids were found. The grass was harvested by cutting manually whenever it reached a length of 12-15 cm in most of the pots. The interval between cuts was 13 to 40 days, becoming greater as the year progressed. A total of eight harvests was taken from soils 1 to 10 and six harvests from soils 11 to 13.

Analysis of grass samples

Grass samples were dried at 90 °C overnight before weighing and milling. A subsample of 0.2 g of milled and re-dried grass was digested with a sulphuric acid/salicylic acid mixture. The digest was de-colourised

with hydrogen peroxide and the P content determined using a Technicon auto-analyser.

RESULTS

Analysis of the TSP products

The range of total P_2O_5 in the products was within the range normally expected for commercial material. One TSP from the UK was somewhat lower in total P_2O_5 than were the other three TSP products but was well above the lower limit (38% P_2O_5) required by EU regulations. There was little difference in the solubility of each TSP in neutral ammonium citrate. Water soluble P_2O_5 , expressed as a percentage of neutral ammonium citrate P_2O_5 , exceeded the 93% EU requirement in the UK-TSP products but not in those from the USA (Table 1).

The water solubility of the US-TSP products, expressed as a percentage of the P_2O_5 soluble in neutral ammonium citrate, was on average 91.6%, much larger than the 80-83% often quoted for US products. Subsequent enquiries showed that in the US, water solubility is calculated as % water-soluble P_2O_5 /% total P_2O_5 , which will inevitably lead to a lower percentage water solubility compared with the EU convention of expressing percentage water solubility relative to the % P_2O_5 soluble in neutral ammonium citrate.

Analysis of the residue sources

Three of the water insoluble components had very similar total amounts of P (expressed as % P_2O_5); but UK-2 had only about half the total P that the other samples contained (Table 1). All four water-insoluble

components contained a small amount (0.5–1.7%) of P₂O₅ which was soluble in water when extracted again after drying (Table 1). This was probably because some water with its dissolved P, remained in the wet insoluble residue after filtering off the bulk of the aqueous solution.

Dry-matter yield and P offtake in the harvested grass

Significant treatment differences were found for all soils, the control giving the lowest cumulative yield or P offtake over the several harvests (Tables 3 and 4).

Relative effectiveness

The relationships between cumulative grass DM yield and amount of P applied in MCP and between cumulative grass P offtake and amount of P applied in MCP were described by quadratic functions fitted by regression. Separate regression equations were derived for every one of the 13 soils. Fit to the data was good with R² > 0.90 for dry-matter yield for all soils except 12 (R² = 0.46) and 13 (R² = 0.54). For P offtake, R² > 0.99 for all soils except 12 (R² = 0.92). Using these equations, the yield or P offtake given by each residue source was equated to an amount of P₂O₅ (M g/pot) applied as MCP. The percentage relative effectiveness (% RE) of the P in the residue source was calculated as M × 100/0.2.

In one case (source US-2 soil 1), the yield given by the residue source (tested at 0.2 g P₂O₅/pot) exceeded that given by the largest amount of P (0.25 g P₂O₅/pot) tested as MCP. As it is not appropriate to extrapolate a response curve beyond the range of the rates of the reference material used, the % RE in this case was restricted to 125%.

Yields were much smaller on the three French soils than on the other soils, partly because there were only six harvests. In addition, there was little response in yield or P offtake to any of the applied P treatments because there were large reserves of readily available soil P. In two cases for yield (source UK-1 soil 13; source UK-2 soil 12) and in four cases in P offtake (source US-1 soil 13; source UK-1 soils 12 and 13; source UK-2 soil 12), the yield or P offtake given by the residue source (tested at 0.2 g P₂O₅/pot) was slightly smaller than the intercept of the regression equation. In these cases, the % RE was set to zero.

When averaged over all 13 soils, % RE of the two US residue sources was much larger than that of the two UK residue sources for both yield and P offtake data (Table 5). There also was a large difference in % RE between the yield and P offtake data, 62 and 46% respectively, when averaged over all four residue sources. This difference arose, in part, due to the difference in the size of the response to MCP from zero to 0.25 g P₂O₅/pot. Yield increased by only 55% but P offtake by twice as much, 112%.

Table 8. *Effect of soil organic carbon on relative effectiveness (% RE)*

Residue source	% RE derived from			
	Yield		P offtake	
	Soil organic carbon			
	<0.9%	>1.0%	<0.9%	>1.0%
US-1	45	78	36	47
US-2	70	92	68	79
Mean	58	85	52	63
UK-1	35	56	17	30
UK-2	40	76	40	45
Mean	38	66	28	38
Overall mean	48	76	40	50

Table 9. *Effect of soil textural class on relative effectiveness (% RE)*

Residue source	% RE derived from					
	Yield			P offtake		
	Soil textural class					
	Sand	Loam	Clay	Sand	Loam	Clay
US-1	63	72	38	36	48	43
US-2	77	90	64	74	78	72
Mean	70	81	51	55	63	58
UK-1	44	54	29	26	30	28
UK-2	64	69	32	43	42	39
Mean	54	62	30	34	36	34
Overall mean	62	71	41	45	50	46

Using the data for the readily available soil P (Table 2), the 13 soils were then grouped as low (less than 30 mg P/l), medium (31–50 mg P/l) and high (more than 51 mg P/l). Averaged over all four residue sources, the % RE in terms of yield was the same on low and medium-P status soils and even larger on the high P soils (Table 6). The average % RE for the two US residue sources in terms of both yield and P offtake was considerably larger than that of the two UK residue sources at all three soil P levels, except in terms of yield on the high P soils.

Using the data for soil pH (measured in water, Table 1), the soils were then grouped as acid (pH less than 6.5), slightly acid to slightly alkaline (pH 6.5–7.5) and alkaline (pH greater than 7.5). Averaged over all four residue sources the % RE in

Table 10. *Effective availability of the P₂O₅ in the four TSP products*

	TSP sample code and source			
	US-1	US-2	UK-1	UK-2
Total P ₂ O ₅ (%)	47.6	48.2	47.0	45.7
Water-soluble P ₂ O ₅ (%)	40.0	41.2	42.5	43.5
% plant availability of P ₂ O ₅ derived from yield				
RE of the water-insoluble residue (%)	63	82	46	59
Available P ₂ O ₅ in water-insoluble residue (%)	4.8	5.7	2.5	1.3
Total effective P ₂ O ₅ (%)	44.8	46.9	45.0	44.8
Effective P ₂ O ₅ as % of total P ₂ O ₅	94	97	94	98
% plant availability of P ₂ O ₅ derived from P offtake				
RE of the water-insoluble residue (%)	42	74	28	41
Available P ₂ O ₅ in water-insoluble residue (%)	3.2	5.2	1.5	0.9
Total effective P ₂ O ₅ (%)	43.2	46.4	44.0	44.4
Effective P ₂ O ₅ as % of total P ₂ O ₅	91	96	92	97

terms of yield and P offtake was much the same on the soils with pH less than 6.5 and with pH 6.5–7.5 (Table 7). The % RE was less on soils with pH greater than 7.5 and this was so for both the US and the UK residue sources. However, at all pH levels the % RE of the US residue sources was appreciably larger than that of the UK sources. It can be concluded that the % RE of these residue sources was not affected by the pH of the soil within the range found in many agricultural soils.

Using the data for % C in Table 1, the 13 soils were grouped into two classes, those with less than 0.9% organic C and those with more than 1% organic C. Averaged over all four residue sources, the % RE derived from yield data was appreciably larger on soils with more organic matter, and a little larger when derived from P offtake data (Table 8). This was unexpected because the experiment was made in pots of soil and visual observation suggested considerable root penetration throughout the whole mass of soil. However, at both levels of soil organic matter and for both yield and P offtake, the % RE of the residue sources from the US was greater than that of those from the UK.

To determine if there was any effect of soil texture, the 13 soils were grouped into three classes, sandy, loamy and clayey on the basis of the percentage of sand, silt and clay. Averaged over all four residue sources, textural class has little effect on % RE except when judged by yield when % RE was appreciably less on the clayey soils (Table 9). This effect, like that of soil organic carbon, was unexpected in a pot experiment. However, for all three soil textural classes, the % RE of the residue sources from the US–TSP products was greater than that of those from the UK–TSP products especially when derived from the yield data.

Effective P concentration in the TSP materials

If some proportion of the P in the water-insoluble component of TSP is as available to a plant as is the P in water-soluble MCP then the 'equivalent water solubility' of the component can be calculated. For example, if TSP contains total P equivalent to 48% P₂O₅ and 40% water-soluble P₂O₅ then there is 8% P₂O₅ in the water-insoluble component. If the % RE of this fraction is 50% then this is equivalent to 4% P₂O₅ in plant available forms. This 4% should be added to the 40% water-soluble P₂O₅ (giving 44% P₂O₅) as a measure of the plant available phosphorus in the immediate/short term. This 44% P₂O₅ can be expressed as a percentage of the total P₂O₅ (48% P₂O₅) giving 91.7% as the percentage effectiveness of the total phosphorus in TSP relative to MCP, a fully water-soluble phosphorus source. This calculation was done for all four residue samples tested on the 13 soils using % RE as derived from the grass dry-matter yield and P offtake data in Table 5. Expressed in this way, between 91 and 98% of the total P₂O₅ in the TSP materials was effective, the range of values was the same for US- and UK-sourced products and there was no systematic difference between them (Table 10).

DISCUSSION

Results of the present study show that although TSP products can vary in the percentage water solubility of the P, the plant availability of the P in the water-insoluble fraction varies also. Thus, the availability to plants of the P in TSP in the short/medium term can be the same irrespective of the percentage water solubility of the P. Similar results were obtained by Mullins *et al.* (1990). They took the water-insoluble

fraction from five TSP products that had been produced from phosphate rocks from Florida, Idaho, Morocco and North Carolina and had water solubilities ranging from 80 to 93%. The effects of these residues were tested, at rates varying from 0 to 80 mg P/kg soil, on sorghum/sudangrass growing in pots in the glasshouse. The water-insoluble residues from the Florida and North Carolina TSP products, which had the least water solubility, increased yields more than did the residues from the Idaho and Morocco products which had the greatest water solubility. Later, Mullins *et al.* (1995) extracted the water-insoluble residue from TSP products made from the same four phosphate rocks and mixed these with MCP to give 'prepared phosphatic fertilizers' with 0, 20, 40, 60 and 80% water solubility. These mixes were then added to

soil of pH 6.4 and the amended soils were cropped with sorghum/sudangrass in pots in the glasshouse. To get 90% of the yield given by MCP, 66, 62, 66 and 52% water solubility was required in the mixes prepared using the water-insoluble residues from TSP made from North Carolina, Morocco, Florida and Idaho phosphate rocks respectively. These results of Mullins *et al.* and in the present study indicate that the effectiveness of TSP with low water solubility will depend on the nature of the impurities in the rock. These results indicate that similar TSPs with water solubility greater than 85% will have equal agronomic effectiveness on a wide range of soils in Europe and therefore give no scientific support to the requirement for a minimum 93% water solubility for TSP to be labelled EC-Type fertilizer.

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