



EQual Poultry litter ash field trials

LIFE10 ENV/UK/176 Task 6.2

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Environment Agency
Horizon house, Deanery Road,
Bristol BS1 5AH
Email EQual@environment-agency.gov.uk
www.environment-agency.gov.uk

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Executive summary

Introduction

The EQual LIFE+ programme aims to promote the re-use and recycling of waste materials whilst protecting human health and the environment. Deriving value from waste materials by turning them back into safe, high quality products is an essential element in the move towards a more circular economy. Offering both economic and environmental benefits if supported and regulated appropriately, waste-derived products improve business resource efficiency and competitiveness, reduce reliance on landfill, and help to conserve virgin raw materials. The Environment Agency is leading the programme with six partners.

As part of the EQual programme, field trials for four waste derived materials were undertaken to improve understanding of the behaviour of these materials in the environment. The evidence base obtained from the trials will support the appropriate use of these materials in place of non-waste materials. Two of the field trials focused on the construction industry (pulverised fuel ash and incinerator bottom ash aggregate), and two on agricultural use (poultry litter ash and paper sludge).

This report provides details and results from the poultry litter ash (PLA) field trials, which aimed to further understanding of the environmental (soil, terrestrial organisms and controlled waters) and human health risks of land application of PLA to agricultural soils. The field trials were carried out by ADAS, in conjunction with Harper Adams University College.

Aims

The aims of the field trials were to:

1. assess the environmental impacts of the application of PLA to agricultural soils;
2. provide data to inform future generic Quantitative Risk Assessment (QRA); and
3. improve understanding of the magnitude of agricultural benefits derived from PLA compared to non-waste-derived alternatives.

Aims 1 and 2 were the primary aims of the field trials.

Methodology

The field trials were undertaken at two existing experimental sites - Harper Adams in Shropshire and Gleadthorpe in Nottinghamshire; both on light textured soils (6-12% clay). The research compared PLA to an 'industry standard' comparator to PLA (Triple Super Phosphate (TSP) and Muriate of Potash (MOP)) and a control (normal agricultural practice).

The comparator (termed the 'P&K treatment') was applied at rates equivalent to the phosphate and potash supplied by the PLA. Each treatment was replicated three times. Spring barley and winter wheat were grown at Gleadthorpe for harvests in 2013 and 2014, respectively. At Harper Adams, winter wheat was grown in 2013 and spring barley in 2014. Topsoil sampling was undertaken in January 2013 to characterise the sites and then in April 2013 and 2014 to evaluate the effect of PLA additions on topsoil properties over two years.

An additional study to evaluate the effect of PLA storage on the environment and the fertiliser properties of PLA (over a c.12 month period) was established in August 2013. Three replicate heaps of PLA (c. 5 t/heap) were constructed in a series of hydrologically isolated bunkers. The volume and chemical composition of leachate draining from the heaps was determined over the 12 month period, together with any changes in the composition of the PLA material using a series of litter bags buried within the heaps.

Results

Two annual applications of PLA resulted in an increase in topsoil extractable P concentrations at Gleadthorpe from 41 mg/l (Index 3) at site characterisation in January 2013 to 60 mg/l (Index 4) in April 2014. This confirmed its value as a P fertiliser, useful for building up soil P levels on low Index soils. This was not apparent at Harper Adams, where soils were already well-supplied with P (with c.85 mg/l extractable P in the topsoil in April 2013 and c.87mg/l extractable P in April 2014; Index 5).

There was no effect of PLA additions on topsoil extractable K concentrations at either site in 2013 and 2014 ($P>0.05$). At Harper Adams extractable K averaged 168 mg/l and 150 mg/l in 2013 and 2014, respectively (Index 2-). At Gleadthorpe extractable K ranged between 60-100 mg/l (Index 1) suggesting additional K was required to maximise yields. However, it should be noted that sandy soils such as those at Gleadthorpe and Harper Adams typically have a low cation exchange capacity. Therefore, these soils are less likely to retain mobile cations like potassium, and consequently often require annual potash applications to maintain crop yields and soil K status.

PLA applications also resulted in a temporary increase in topsoil sodium concentrations ($P<0.05$) shortly after the first application at both sites in 2013. Although higher than the control and P&K treatment plots, concentrations were still at the low end of the range of reported values for soils in England and Wales. Concentrations declined over the subsequent 12 months such that by April 2014 there was no difference between the treatments, despite a second application of PLA in Autumn 2013, most likely due to over-winter leaching.

In the storage study, there was a significant decline in total potash, boron, molybdenum, selenium and vanadium ($P<0.05$), representing a loss of valuable major and trace nutrients

Conclusions

The results demonstrated that PLA is beneficial as a fertiliser, and indicated that PLA can be used for building up soil P levels on low Index soils.

The study also demonstrated that the short-term annual application of PLA to agricultural soils growing cereal crops presented no significant environmental risks. No significant negative effect of the two annual PLA applications on topsoil aggregate stability, microbial biomass, the measured soil chemical properties, uptake of potential contaminants of concern (COPCs) or crop yields were observed over the timescales of the field trials.

The storage study showed that there was a loss of potash (which declined by c.15%) and other valuable trace nutrients (specifically boron, molybdenum and selenium) over the duration of the study. The storage heaps also generated a highly reactive alkaline (c. pH 12) leachate containing elevated concentrations of soluble P which would be highly detrimental if it entered surface water bodies in an un-diluted form.

The study therefore demonstrated the importance of adhering to the Quality Protocol with respect to storing PLA materials in temporary field heaps to prevent surface water pollution (i.e. PLA should be stored in a dry environment in order to prevent the generation of potentially harmful leachate together with a loss of valuable nutrients).

The field trials have improved understanding of the environmental impacts and agricultural benefits of the application of PLA to agricultural soils; and have provided useful data and understanding to inform future QRAs. The objectives for the EQual PLA field trials have therefore been achieved.

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Introduction

1.1. Background

In the United Kingdom, millions of tonnes of waste-derived materials are landfilled every year. Removal of appropriate waste-derived materials from landfill if they can be beneficially recycled to land has the potential to provide benefits in terms of the sustainable use of plant nutrients, potentially reducing the need for manufactured fertilisers. However, it is essential that the application (agricultural or otherwise) is truly beneficial and is not more harmful to the environment (i.e. soil, water and air quality) or human health than the non-waste material it replaces.

The EQual LIFE+ programme aims to promote the re-use and recycling of waste materials whilst protecting human health and the environment. Deriving value from waste materials by turning them back into safe, high quality products is an essential element in the move towards a more circular economy. Offering both economic and environmental benefits, if supported and regulated appropriately, waste-derived products improve business resource efficiency and competitiveness, reduce reliance on landfill, and help to conserve virgin raw materials.

The Environment Agency is leading the programme with six partners: Rijkswaterstaat (the Netherlands' Ministry of Infrastructure and the Environment), The Chartered Institution of Wastes management, Organics Recycling Group, Environmental Services Association, Northern Ireland Environment Agency and Energy UK.

As part of the EQual programme, field trials were carried out for four waste derived materials to improve understanding of the behaviour of these materials in the environment. The evidence base obtained from the trials will support the appropriate use of these materials in place of non-waste materials

Two of the field trials focused on the construction industry (pulverised fuel ash and incinerator bottom ash aggregate), and two on agricultural use (poultry litter ash and paper sludge). This document reports the poultry litter ash (PLA) field trials. The field trials were carried out by ADAS UK Ltd and Harper Adams University College.

1.2. PLA field trials

PLA is a waste-derived material from the combustion of predominately poultry litter (straw, woodchip and sawdust) which has been designated for use as a phosphate and potash fertiliser. The Quality Protocol for PLA sets out the end of waste criteria for the production and use of PLA as a phosphate (P_2O_5) and potash (K_2O) fertiliser in the UK (EA & WRAP, 2012), enabling PLA compliant with this Quality Protocol to be used as a non-waste.

Previous research on PLA has largely focused on the P_2O_5 and K_2O fertiliser replacement benefits of such applications, with limited research on the impact of applications to the wider environment (i.e. water, crop and soil quality) and human health.

PLA field trials were carried out to further understanding of the environmental (soil, terrestrial organisms and controlled waters) and human health risks of land application of PLA to agricultural soils compared with non-waste-derived alternatives.

The aims of the field trials were to:

1. Assess the environmental impacts of the application of PLA to agricultural soils;
2. Provide data to inform future generic Quantitative Risk Assessment (QRA)*; and
3. Improve understanding of the magnitude of agricultural benefits derived from PLA compared to non waste-derived alternatives.

*QRA does not form part of this project.

Aims 1 and 2 were the primary aims of the field trials.

The objectives of the field trials were to answer the following specific research questions:

1. With respect to specific determinands (Section 2), how do physical and chemical properties of soil and pore water change over time in:
 - a. Control plots;
 - b. Plots with application of PLA; and
 - c. Plots with application of 'industry standard' comparator to PLA.
2. How do physical and chemical properties of soil and pore water in plots with application of PLA compare with those in plots with industry standard alternatives/equivalents and control plots?
3. To what extent are COPCs taken up by crops grown with application of PLA?
4. How does uptake of COPCs by crops grown with application of PLA compare to uptake by crops grown with application of industry standard alternatives/equivalents and in control plots?
5. How does the total soil microbial biomass change over time in soils with application of PLA?
6. How does total soil microbial biomass in soils with application of PLA compare with total soil microbial biomass with application of industry standard alternatives/equivalents and in control plots?
7. How do storage durations affect key properties of the PLA (e.g. nutrient content, pH, pathogens)?
8. How do the yields of crops grown with application of PLA compare with those grown with application of industry standard alternatives/equivalents and in control plots?
9. How do the yield benefit-costs of PLA compare to those of industry standard alternatives/equivalents?

Questions 1 to 7 were the primary objectives.

Methodology

2.1. Field trials sites

The research was undertaken at two existing experimental sites at Harper Adams (sandy loam textured soil) and ADAS Gleadthorpe (loamy sand textured soil) sites (Table



0-1 &



a) Harper Adams; spring barley June 2014

b) Gleadthorpe – spring barley; June 2013

Figure 0-1).

Table 0-1. Field trials sites

Site	Soil properties ¹			Cropping rotation ²	
	Texture	Organic matter (%)	Bulk density (g/cm ³)	2012/13	2013/14
1. Harper Adams	Sandy loam (12% clay)	2.7	1.23	WW	SB
2. Gleadthorpe	Loamy sand (6% clay)	2.4	1.42	SB	WW

¹Topsoil properties measured in 2001 (Bhogal *et al.*, 2009)

²WW = Winter wheat; SB = Spring barley



b) Harper Adams; spring barley June 2014



b) Gleadthorpe – spring barley; June 2013

Figure 0-1. Field trials sites

2.2. Experimental treatments and design

The trials comprised of three treatments:

- Control – normal agricultural practice;
- PLA – normal agricultural practice plus 700 kg/ha PLA;
- An ‘industry standard’ comparator to PLA – normal agricultural practice plus Triple Super Phosphate (TSP) and Muriate of Potash (MOP) to match the P₂O₅ and K₂O loadings of the PLA); termed the ‘P&K’ treatment; and

There were 3 replicates of each treatment and control arranged in a randomised blocks design at Harper Adams and fully randomised design at Gleadthorpe (Figure 0-2). Plots were 6 x 10m at Harper Adams and 5 x 15m at Gleadthorpe. The treatment plots were embedded within a pre-existing experimental layout at each of the sites.

a) Harper Adams (randomised blocks) – treatments discussed in this report are highlighted in grey

<i>Block 1</i>	<i>Block 2</i>	<i>Block 3</i>
Historic PS	PLA	Fresh PS
Control	Fresh PS	Historic PS
PLA	P&K	Control
Fresh PS	Control	P&K
P&K	Historic PS	PLA

b) Gleadthorpe (fully randomised) – treatments discussed in this report are highlighted in grey

Fresh PS	P&K	PLA	P&K	Historic PS	Control
Fresh PS	PLA	P&K	PLA	Control	
	Fresh PS	Historic PS	Control	Historic PS	

* note: the PLA and P&K treatments were embedded within a pre-existing experimental layout

Figure 0-2. Schematic of the experimental layout at each site

2.3. Site characterisation (baseline) sampling

Topsoil (0-15cm depth) samples were taken in January 2013 from each of the plots, in order to characterise the soils prior to treatment application and determine the baseline against which to assess results. Samples were taken using a hand-held corer and comprised of c.20 cores taken at intervals across each plot and bulked together to give a single soil sample from each plot (see

Appendix 1: method statement for baseline soil sampling). Table 0-2 details the analyses undertaken on each of the soil samples.

Additional soil samples were taken for the determination of soil microbial biomass carbon (C) and nitrogen (N), using the chloroform incubation methodology (see Appendix 1: method statement for microbial biomass analysis; Jenkinson & Powelson, 1976), and aggregate stability using the dispersion ratio method on 5-30mm soil aggregates (see Appendix 1: method statement for aggregate stability; Anon, 1982).

Table 0-2. Topsoil analysis suite

Property	Treatment plots
Total Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Ag, Na, Sr, Tl, Sn, Ti, V and Zn	All
Hexavalent Cr (CrVI)	All
Dioxins, Furans & Dioxin-like PCBs	PLA, P&K & control - Gleadthorpe only
pH	All
Olsen extractable P	All
Ammonium nitrate extractable K & Mg	All
Microbial biomass C and N	All
Aggregate stability	All

2.4. Material application

Application of PLA and the industry standard comparator to PLA (TSP and MOP) was carried out in February 2013 and again in Autumn 2013. PLA was applied at a rate of 700 kg/ha, TSP at 185 kg/ha (supplying 84kg/ha P₂O₅) and MOP at 140 kg/ha (supplying 84 kg/ha of K₂O). All applications were carried out by hand. Following application, the treatments were incorporated by ploughing at Gleadthorpe (plough depth: c.20cm) in both February and November 2013. At Harper Adams, the treatments were top-dressed to the growing winter wheat crop in February 2013 and incorporated by disc (to a depth of c.15cm) in October 2013, (see Appendix 1: method statement for PLA, TSP and MOP applications).

All treatments, including the control received recommended rates of manufactured fertiliser nutrients based on the 'Fertiliser Manual, RB209 (Defra, 2010) in order to minimise potential confounding interactions and ensure (as far as was practically possible) that no major nutrient limited plant growth. At Gleadthorpe this included the application of 5 t/ha lime as the pH across the site was c.6.0 in December 2012 ahead of the planting of spring barley, a crop sensitive to soil acidity (MAFF, 1983). Details of the fertiliser products used and rates of application are given in Table 0-3, with a full diary of activities at the field trials sites given in Appendix 2.

The PLA was supplied by the Steering Group. Material was stored and used in accordance with the PLA Quality Protocol (EA & WRAP, 2012).

Table 0-3. Fertiliser policy at each of the field trials sites

Fertiliser ¹	Gleadthorpe		Harper Adams	Spring barley (2014)
	Spring barley (2013)	Winter wheat (2014)	Winter wheat (2013)	
Lime	5 t/ha	-	-	-
Nitrogen- N ²	110 kg/ha: 40 kg/ha as AS 70 kg/ha as AN	160 kg/ha: 40 kg/ha as AS 120 kg/ha as AN	180 kg/ha: 40 kg/ha as AS 140 kg/ha as AN	110 kg/ha N as AN
Potash -K ₂ O ³	100 kg/ha as MOP	100 kg/ha as MOP	75 kg/ha as MOP	35 kg/ha as MOP
Sulphur-SO ₃ ⁴	50 kg/ha as AS	50 kg/ha as AS	40 kg/ha as AS	50 kg/ha as Kieserite

¹There was no requirement for phosphate (P) fertilisers at either of the sites due to high levels of extractable P within the topsoil at the outset of the trials (c.55 mg/l P, Index 4 at Gleadthorpe and c.80 mg/l P, Index 5 at Harper Adams); ² Nitrogen supplied as a combination of Ammonium Sulphate (AS) and Ammonium Nitrate (AN); ³Potash supplied as Muriate of Potash (MOP); ⁴Sulphur supplied as either Ammonium Sulphate (AS) or Magnesium Sulphate (Kieserite).

2.5. Analysis of the applied materials

Samples of the materials applied at each site were taken to provide a cross-check to the compositional data provided with the materials, to enable material variability to be taken into account. Triplicate samples of the PLA from each site, were taken in Spring 2013 and Autumn 2013, together with a single sample of the TSP and MOP applied at Gleadthorpe in Spring 2013.

Table 0-4 details the analyses undertaken on the applied materials.

Table 0-4. Material analysis suite

Property	Treatment plots
Dry matter & pH	PLA
Total Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Ag, Na, Sr, Tl, Sn, Ti, V and Zn	PLA
Hexavalent Cr (CrVI)	PLA
Dioxins, Furans & Dioxin-like PCBs	TSP & MOP

2.6. Soil sampling and analysis

Topsoil samples were taken in April 2013, c.2 months after the first annual application of PLA, and again in April 2014, c.6 months following the second annual application of PLA. At each sampling, topsoil samples (0-15cm) were taken from each plot (see method statement for annual soil sampling; Appendix 1) for the analyses detailed in Table 0-2. As at site characterisation the original proposal also intended to analyse the pore water extracted from the soil. However even though samples were taken when the soil was close to field capacity, water retention on these sandy soils is very low and insufficient pore water could be extracted for any meaningful analyses to be performed.

Additional soil samples were taken for the determination of soil microbial biomass carbon (C) and nitrogen (N), using the chloroform incubation methodology (Jenkinson & Powlson, 1976), and aggregate stability using the dispersion ratio method on 5-30mm soil aggregates (Anon, 1982), as described above and in Appendix 1.

2.7. Crop yields and grain analysis

Crop yields were determined in August 2013 and 2014 at both sites using a plot combine (see method statement for grain/seed yield and analysis; Appendix 1), with samples of the grain analysed for total Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Ag, Na, Sr, Tl, Sn, Ti, V and Zn.

2.8. Storage study

An additional study was carried out to evaluate the effect of PLA storage on the environment and the properties of PLA. Replicated field heaps (3 replicates per material) of PLA were established at Gleadthorpe in August 2013 (Figure 0-3). Each heap comprised of c. 5 tonnes material established in a series of hydrologically isolated, sloping concrete bunkers. Leachate from each heap was collected at the lowest corner of the bunkers using a short length of perforated plastic drainage pipe to direct the leachate into three individual collection tanks.

During construction of the heaps, triplicate samples were taken for analysis and four 'litter' bags, each containing c.2 kg of the PLA material, were buried at known separate locations within each heap. These were retrieved after 1, 3 and 6 months and at the end of the storage period and analysed as detailed in

Table 0-4. Measurements of the quantity of leachate from each storage heap were taken and related to rainfall volumes.

Samples were taken from the collection tanks on a monthly basis and analysed for total N, ammonium-N, nitrate-N, Total P, orthophosphate-P, BOD, pH, Total Al, Sb, As, Ba, Be, Cd, Cr, hexavalent Cr, Co, Cu, Fe, Pb, Mn, Hg, Mo, Ni, Se, Ag, Te, Tl, Sn, Ti, U, V and Zn. See Appendix 3 for a detailed method statement covering the storage study.



Figure 0-3. Construction of the PLA storage heaps

2.9. Statistical analysis

The results from the field study were analysed using conventional one way analysis of variance (ANOVA), assuming a fully randomised design at Gleadthorpe and a randomised blocks design at Harper Adams. All analyses were performed using Genstat version 12 (VSN International Ltd, 2010) and evaluated whether there were any statistically significant ($P < 0.05$) treatment effects on the soil chemical, biological and physical properties, crop yields and uptake of COPCs.

Where significant differences were seen, post-hoc testing was undertaken to evaluate which treatment means were different from each other using a Duncan's multiple range test (in Genstat 12). Changes in soil properties over the course of the trials (from baseline sampling in January 2013 to April 2014) were evaluated using ANOVA, with time (3 levels: January 2013, April 2013 & April 2014) and treatment (3 levels: control, PLA & P&K) as factors.

Potential changes in the composition of stored PLA and its leachate during the c.12 month storage period were evaluated using both ANOVA, to determine whether there were any statistically significant changes over time ($P < 0.05$), and regression, to evaluate the magnitude and direction of any changes in composition (using Genstat version 12).

Results

3.1. Site baseline characterisation

Characterisation of the field trials plots was undertaken in January 2013 prior to treatment application in order to determine the baseline against which to assess the results (Table 0-1)

Table 0-1. Site baseline soil characterisation (treatment means), January 2013

Determinand	Units	Gleadthorpe				Harper Adams			
		Con	PLA	P&K	P ¹	Con	PLA	P&K	P ¹
Conductivity		2093 ^b	1987 ^a	1997 ^a	0.008	2120	2043	2090	NS (0.13)
pH	unit	5.69	5.60	5.65	NS (0.77)	6.11	6.76	6.79	NS (0.44)
Extractable P	mg/l	53.6	41.3	44.4 ²	NS (0.30)	79.4 ²	89.4	101	NS (0.27)
Extractable K	mg/l	68.7	62.7	63.9	NS (0.80)	162	185	154	NS (0.74)
Extractable Mg	mg/l	67.8 ^b	34.8 ^a	36.1 ^a	<0.001	57.1	55.5	56.5	NS (0.98)
Aluminium	mg/kg dm	5337	5217	5187	NS (0.96)	11333	12033	11567	NS (0.78)
Antimony	mg/kg dm	<10.0	<10.0	<10.0	NS (1.0)	<10.0	<10.0	<10.0	NS (1.0)
Arsenic	mg/kg dm	4.90	4.62	4.60	NS (0.66)	6.19	6.48	6.01	NS (0.65)
Barium	mg/kg dm	30.7	29.1	59.8	NS (0.49)	64.3	104	62.0	NS (0.37)
Beryllium	mg/kg dm	0.34	0.32	0.33	NS (0.91)	0.47	0.60	0.48	NS (0.31)
Boron	mg/kg dm	3.60	3.96	3.61	NS (0.71)	8.45	15.2	10.0	NS (0.42)
Cadmium	mg/kg dm	<0.25	<0.25	<0.25	NS (1.0)	<0.25	<0.25	<0.25	NS (1.0)
Calcium	mg/kg dm	908	758	750	NS (0.48)	2237	2917	2927	NS (0.52)
Chromium	mg/kg dm	7.25	7.20	6.95	NS (0.90)	13.4	14.4	13.3	NS (0.72)
Chromium VI	mg/kg dm	<0.30	<0.30	<0.30	NS (1.0)	<0.30	<0.30	<0.30	NS (1.0)
Cobalt	mg/kg dm	1.97	1.77	1.76	NS (0.38)	3.79	4.13	3.70	NS (0.49)
Copper	mg/kg dm	5.56	5.31	5.14	NS (0.77)	12.5	13.8	12.8	NS (0.69)
Iron	mg/kg dm	8407	7797	7847	NS (0.65)	11267	11967	11133	NS (0.65)
Lead	mg/kg dm	16.5	16.9	15.1	NS (0.70)	17.4	18.3	16.4	NS (0.44)
Lithium	mg/kg dm	7.45	6.97	7.08	NS (0.68)	14.7	16.0	14.5	NS (0.66)
Magnesium	mg/kg dm	783	748	734	NS (0.88)	2393	2573	2403	NS (0.73)
Manganese	mg/kg dm	204 ^b	141 ^a	152 ^{ab}	0.05	281	295	260	NS (0.63)
Mercury	mg/kg dm	< 2.00	< 2.00	< 2.00	NS (1.0)	< 2.00	< 2.00	< 2.00	NS (1.0)
Molybdenum	mg/kg dm	< 2.00	< 2.00	< 2.00	NS (1.0)	< 2.00	< 2.00	< 2.00	NS (1.0)
Nickel	mg/kg dm	4.33	4.57	4.33	NS (0.75)	9.85	10.9	9.70	NS (0.55)
Phosphorus	mg/kg dm	659	563	554	NS (0.49)	1060	1207	1073	NS (0.32)
Potassium	mg/kg dm	704	679	660	NS (0.84)	2137	2287	2190	NS (0.84)

Determinand	Units	Gleadthorpe				Harper Adams			
		Con	PLA	P&K	P ¹	Con	PLA	P&K	P ¹
Selenium	mg/kg dm	<1.00	<1.00	<1.00	NS (1.0)	<1.00	<1.00	<1.00	NS (1.0)
Silver	mg/kg dm	<10.0	<10.0	<10.0	NS (1.0)	<10.0	<10.0	<10.0	NS (1.0)
Sodium	mg/kg dm	29.4	31.6	31.6	NS (0.77)	62.0	87.1	68.5	NS (0.18)
Strontium	mg/kg dm	<5.00	5.16	<5.00	NS (1.0)	8.33	10.6	9.56	NS (0.47)
Thallium	mg/kg dm	<3.00	<3.00	<3.00	NS (1.0)	<3.00	<3.00	<3.00	NS (1.0)
Tin	mg/kg dm	<20.0	<20.0	<20.0	NS (1.0)	<20.0	<20.0	<20.0	NS (1.0)
Titanium	mg/kg dm	68.0	65.1	66.1	NS (0.89)	92.5	105	97.8	NS (0.07)
Vanadium	mg/kg dm	11.0	9.83	10.2	NS (0.51)	17.2	18.3	16.8	NS (0.67)
Zinc	mg/kg dm	29.8	26.1	24.8	NS (0.46)	60.3	69.9	58.7	NS (0.27)
Sum 7 PCBs	mg/kg dm	<0.01	<0.01	<0.01	NS (1.0)	nd	nd	nd	-
Sum 12 PCBs TEQ	ng/kg dm	<0.01	<0.01	<0.01	NS (1.0)	nd	nd	nd	-
Dioxins & furans TEQ	ng/kg dm	5.27	6.05	5.50	NS (0.35)	nd	nd	nd	-
Biomass C	mg/kg	69	56	50	NS (0.18)	168	197	208	NS (0.12)
Biomass N	mg/kg	14	12	13	NS (0.39)	31	38	37	NS (0.16)
Aggregate stability ³	% w/w	14	16	19	NS (0.07)	8	7	10	NS (0.64)

¹Statistical analysis undertaken using ANOVA (data normally distributed); numbers in brackets indicate the P statistic; ^{a,b}Different letters between columns indicate significant differences between treatments for a particular site at $P < 0.05$; ²Mean of 2 replicate plots; ³Aggregate stability measured by the dispersion ratio (ratio of silt and clay suspended by mild slaking forces expressed as a % of the total silt and clay content); Ratios in the range 6-10% suggest the soil is 'stable', 11-15% indicate that the soil is 'fairly stable', while ratios in the range 16-25% suggest the soil is 'somewhat unstable' (Anon, 1982).

At Gleadthorpe, there was significant background variation ($P < 0.01$) in topsoil conductivity, extractable magnesium (Mg) and total manganese (Mn), with the untreated control plots higher than the untreated PLA and P&K treatment plots. This background variation was taken into account when considering the results of the topsoil sampling undertaken in April 2013 and 2014. There was no significant background variation in soil properties at Harper Adams.

The Index soil system (Defra, 2010) classifies soils according to their soil P and K (and Mg) status and identifies target indices at which the supply of crop-available phosphate or potash is considered to be sufficient to maximise crop yields. For arable crops and grass these are Index 2 for P (16-25 mg/l Olsen-extractable P) and Index 2- for K (121-180 mg/l ammonium-nitrate extractable K), and fertiliser recommendations aim to maintain soils at these levels (Defra, 2010). Background soil extractable P concentrations were high at both sites, with c.55 mg/l P, Index 4 at Gleadthorpe and c.80 mg/l P, Index 5 at Harper Adams.

As a result, no additional phosphate fertilisers were required as part of the fertiliser policy for each site (Table 0-3). Extractable K concentrations were at Index 1 (c.70 mg/l K) at Gleadthorpe indicating additional potash fertiliser was required (Table 3) and at Index 2- (160 mg/l) at Harper Adams, where only a maintenance application of potash was required (to balance offtake of K in the crop; Table 0-3).

3.2. Composition of the applied materials

Table 0-2 gives details of selected chemical properties of the PLA applied at both sites. There was a slight discrepancy in P₂O₅ and K₂O supplied from the PLA and fertiliser equivalents. This arose because the rates of TSP and MOP were calculated based on analysis supplied with the PLA (12% P₂O₅ and K₂O i.e. 84 kg/ha of each nutrient @ 700 kg/ha), rather than analysis of the PLA that was spread.

Table 0-2. Chemical composition of the PLA (treatment means)

Determinand	Units	Gleadthorpe		Harper Adams		Maximum permitted value ¹
		PLA Feb 13	PLA Oct 13	PLA Feb 13	PLA Oct 13	
Dry matter	%	90.7	90.2	90.3	90.1	
pH	unit	12.6	12.6	12.6	12.6	
Phosphate (P ₂ O ₅)	kg/t fw	202	188	196	185	
Potash (K ₂ O)	kg/t fw	168	177	165	179	
Magnesium (MgO)	kg/t fw	60.2	53.8	58.5	53.6	
Calcium	kg/t fw	131	119	129	119	
Aluminium	mg/kg dm	4173	3240	3960	3223	
Antimony	mg/kg dm	4.20	2.72	4.19	2.98	
Arsenic	mg/kg dm	8.37	10.3	8.47	11.0	17
Barium	mg/kg dm	232	176	218	173	
Beryllium	mg/kg dm	0.42	0.34	0.41	0.34	
Boron	mg/kg dm	117	123	116	124	
Cadmium	mg/kg dm	1.29	1.09	1.19	1.14	3
Chromium	mg/kg dm	22.3	21.6	26.0	21.8	31
Chromium VI	mg/kg dm	< 0.30	<0.6	< 0.30	<0.6	
Cobalt	mg/kg dm	5.37	4.78	5.20	4.69	11
Copper	mg/kg dm	477	478	496	478	596
Iron	mg/kg dm	5920	4520	5860	4640	
Lead	mg/kg dm	127	114	128	121	244
Lithium	mg/kg dm	7.70	8.25	7.14	8.16	
Manganese	mg/kg dm	3220	2763	3153	2857	3500
Mercury	mg/kg dm	< 0.20	<0.2	< 0.20	<0.2	0.5
Molybdenum	mg/kg dm	33.2	32.5	32.6	32.4	45
Nickel	mg/kg dm	17.0	14.6	17.1	14.4	24
Selenium	mg/kg dm	3.83	4.75	3.89	4.87	11
Silver	mg/kg dm	< 1.00	<1	< 1.00	<1	
Sodium	mg/kg dm	32167	31533	31800	32133	
Strontium	mg/kg dm	244	217	233	215	
Thallium	mg/kg dm	1.14	1.58	1.17	1.55	
Tin	mg/kg dm	2.89	3.08	2.97	3.30	
Titanium	mg/kg dm	372	338	371	333	
Vanadium	mg/kg dm	18.2	16.1	18.4	16.0	20
Zinc	mg/kg dm	2057	1340	2073	1353	2063

¹Maximum compositional values for trace elements in PLA as a fertiliser (EA&WRAP, 2012)

Actual analysis of the PLA following spreading in both Spring and Autumn 2013 indicated higher contents (c.18% P₂O₅ and 17% K₂O Table 6), such that 130-140 kg/ha/yr P₂O₅ and 115-125 kg/ha/yr K₂O was applied. This was c.45-55 kg/ha/yr and 30-40 kg/ha/yr higher than the respective amounts of P₂O₅ and K₂O applied to the comparator P&K treatment.

This additional P and K on the PLA treatment was unlikely to impact on crop yields, as both sites were well-supplied with P (Index 4-5; Table 0-1), and potash fertiliser (MOP) was routinely applied to all treatments as part of the site's normal fertiliser policy (Table 0-3). However, this additional P and K input to the PLA treatment was taken into account when considering the soil analysis results from the April 2013 and 2014 samplings.

The concentrations of trace elements within the PLA were within the maximum compositional limits set by the Quality Protocol (EA & WRAP, 2012), Table 0-2, although zinc concentration at Gleadthorpe in Feb 2013 was very close to the maximum permitted value.

The TSP and MOP applied at Gleadthorpe were also analysed for organic contaminants (PCBs, dioxins and furans). PCB concentrations were below the limits of detection and dioxins and furans concentrations were low at <2 ng/kg dm TEQ (Table 0-3). For context, the Quality Protocol for PLA gives a maximum compositional dioxin concentration of 20 ng/kg TEQ).

Table 0-3. Chemical composition of the TSP and MOP applied at Gleadthorpe in February 2013 (treatment means)

	Units	TSP Feb 13	MOP Feb 13
Sum 7 PCBs	mg/kg dm	<0.01	<0.01
Sum 12 PCBs TEQ	ng/kg dm	<0.01	<0.01
Dioxins and furans TEQ	ng/kg dm	1.83	1.64

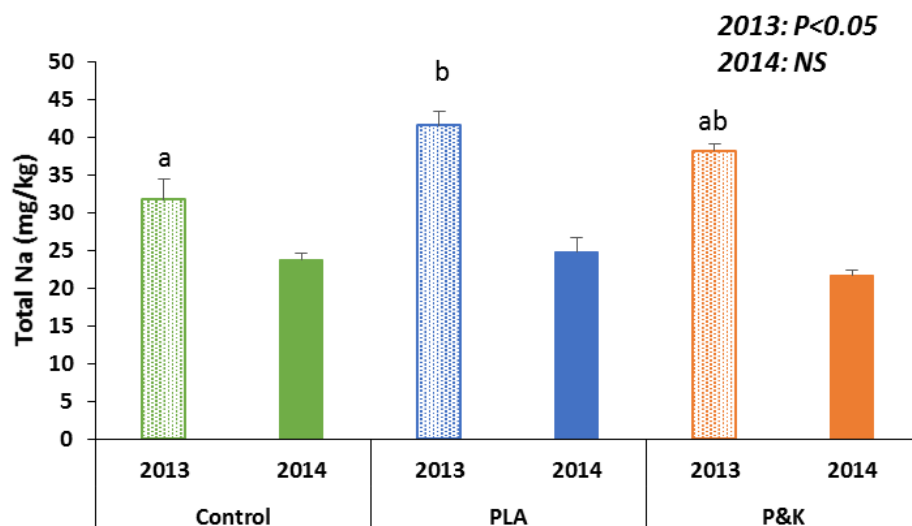
3.3. Effect of PLA on topsoil properties

There was very little effect ($P>0.05$) of PLA or the equivalent manufactured fertiliser P&K treatment on topsoil chemical properties approximately 2 months after application (April 2013; Appendix 4), except for topsoil sodium ($P<0.05$ at both sites; Figure 0-1 and Figure 0-2) and strontium concentrations ($P<0.05$ at Harper Adams only; Figure 0-3 and Figure 0-4), which were higher where PLA had been applied. This was related to elevated concentrations of these elements within the PLA material (Table 0-2), although topsoil concentrations were still at the low end of the range of reported values for soils in England and Wales, which average 79 mg/kg (range: 4.5->1000 mg/kg) for Strontium and 0.32% or 3200 mg/kg (range: 0.074-7.4%) for sodium (Rawlins *et al.*, 2012).

By the second annual sample in April 2014, these differences in topsoil sodium and strontium concentrations were no longer apparent ($P>0.05$). It is therefore likely that the elevated concentrations measured in 2013 were due to the presence of PLA material within the soil sample, which was taken c.2 months after application, and at Harper Adams was top-dressed onto the growing crop, without soil incorporation.

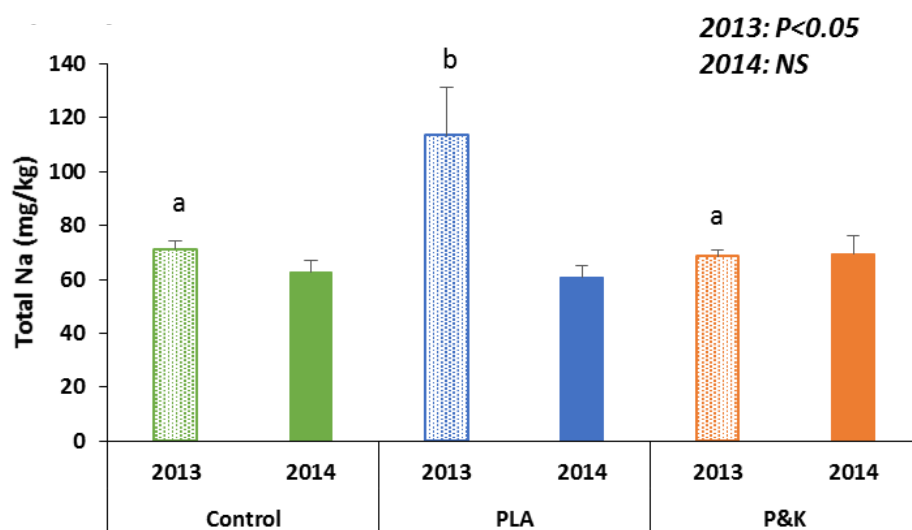
In 2014, the PLA was incorporated into the soil at both sites and there was a much longer period (c.6 months) between PLA application and topsoil sampling. This, together with over-winter rainfall, would have enabled the dissolution and equilibration of sodium and strontium from the applied PLA within the topsoil.

Statistical analysis was carried out of the changes in soil properties over time from baseline sampling in January 2013 to the sampling in April 2014 (following two annual treatment applications). The results suggested this pattern of elevated concentrations shortly after application in April 2013 falling to background concentrations in April 2014 was also apparent for total and extractable potassium, total beryllium, iron and titanium at Gleadthorpe and pH at Harper Adams ($P<0.05$ for change over time; statistical results presented in Appendix 4), although the differences between treatments were inconsistent.



^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P < 0.05$ as determined by ANOVA and post-hoc Duncans analysis; i.e. the PLA^b was significantly different to the control^a, but not the P&K treatment^{ab}; NS=no significant differences in 2014 ($P > 0.05$); changes over time were significant at $P < 0.01$.

Figure 0-1. Topsoil sodium concentrations measured in April 2013 and 2014: Gleadthorpe



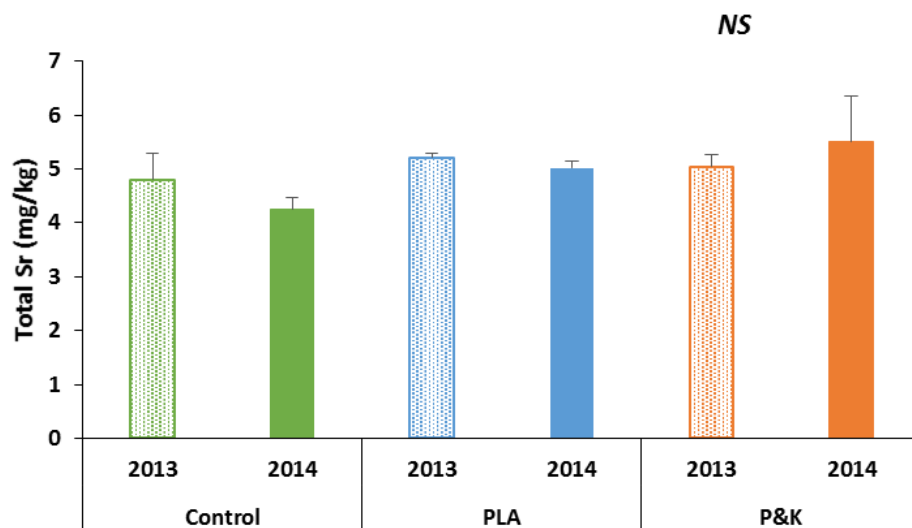
^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P < 0.05$ as determined by ANOVA and post-hoc Duncans analysis; i.e. at Harper Adams in 2013, the PLA^b was significantly different to the control^a, and the P&K treatment^a; NS=no significant differences in 2014 ($P > 0.05$); changes over time were significant at $P < 0.01$.

Figure 0-2. Topsoil sodium concentrations measured in April 2013 and 2014: Harper Adams

There was no significant effect of PLA additions on topsoil extractable potassium (K) concentrations at either site in 2013 or 2014 ($P > 0.05$; Figure 0-5 and Figure 0-6), although the P&K treatment at Gleadthorpe did result in an increase in extractable K in April 2013 ($P < 0.01$), but not April 2014 ($P < 0.001$ for change over time; Appendix 4), suggesting some of the MOP may have been present in the soil sample in April 2013 (c.2 months following application).

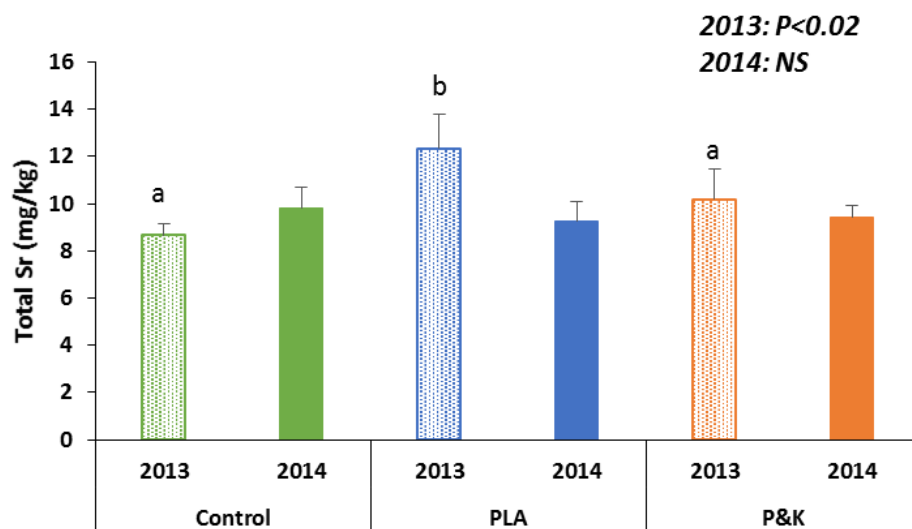
At Gleadthorpe, extractable K ranged between 60-100 mg/l (Index 1) suggesting additional K was required to maximise yields. Concentrations were higher at Harper Adams, where extractable K averaged 168 mg/l and 150 mg/l in 2013 and 2014, respectively (Index 2-), with no difference between treatments. The higher K loading on the PLA treatment, discussed in the material analysis section above, had no impact on topsoil extractable or total K levels.

These results suggest that all the K supplied with the treatments (both the PLA and MOP) was either utilised by the crop (particularly at Gleadthorpe where the soils were low in extractable K) or was leached. Sandy soils such as those at Gleadthorpe and Harper Adams typically have a low cation exchange capacity. They are therefore less likely to retain mobile cations like potassium, and consequently often require annual potash applications to maintain crop yields and soil K status.



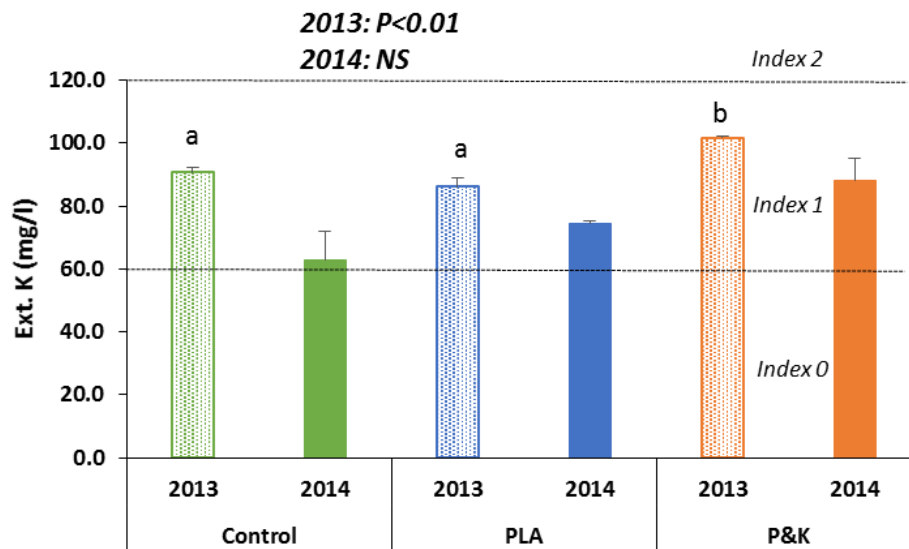
^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P < 0.05$ as determined by ANOVA and post-hoc Duncans analysis; i.e. NS=no significant differences at Gleadthorpe ($P > 0.05$)

Figure 0-3. Topsoil strontium concentrations measured in April 2013 and 2014: Gleadthorpe



^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P < 0.05$ as determined by ANOVA and post-hoc Duncans analysis; i.e. at Harper Adams in 2013, the PLA^b was significantly different from the control^a and the P&K treatment^a; NS=no significant differences in 2014 ($P > 0.05$)

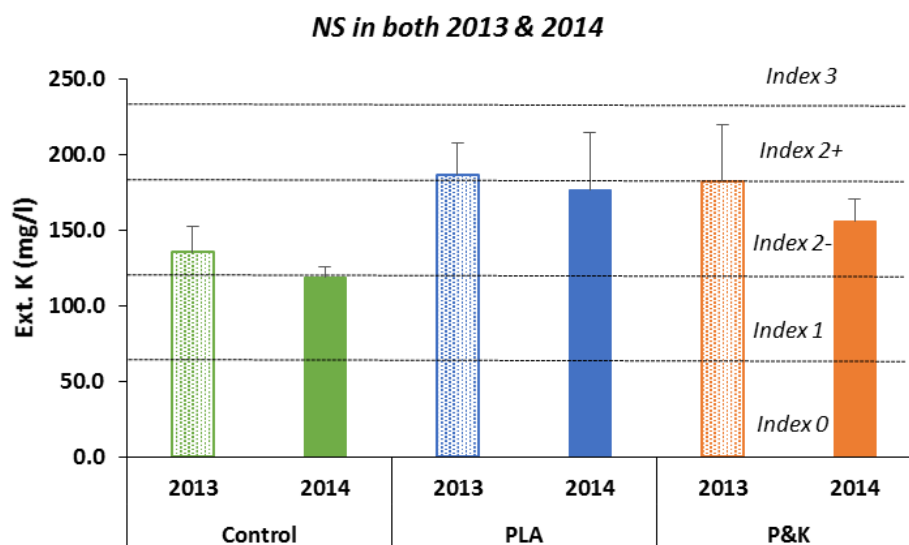
Figure 0-4. Topsoil strontium concentrations measured in April 2013 and 2014: Harper Adams



^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P < 0.05$ as determined by ANOVA and post-hoc Duncans analysis; i.e. at Gleadthorpe in 2013, the P&K treatment^b was significantly different to the control^a and PLA treatments^a; NS=no significant differences in 2014

---- Horizontal lines show where the results fall within the soil index classification system (Defra, 2010) and demonstrate that the soils at Gleadthorpe required additional K fertiliser to maximise crop yields (Index 1).

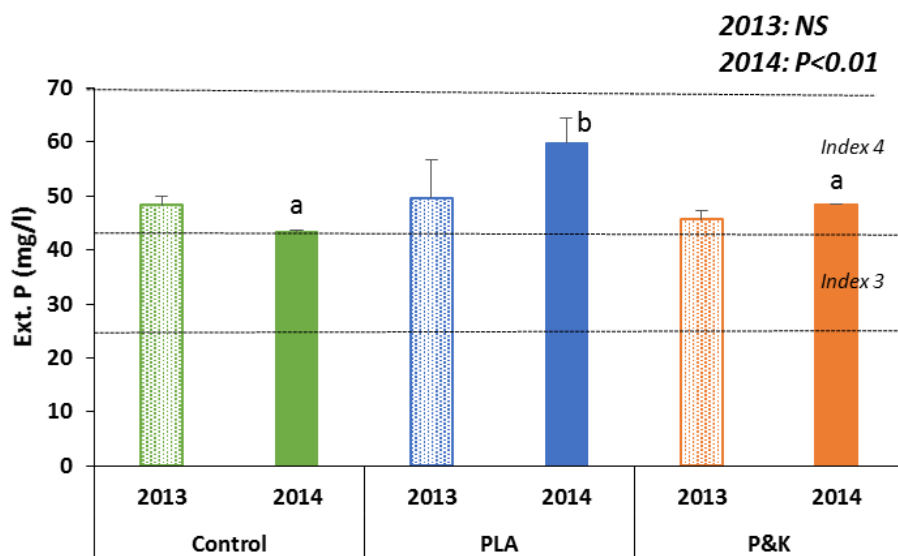
Figure 0-5. Topsoil potassium concentrations measured in April 2013 and 2014: Gleadthorpe



^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P < 0.05$ as determined by ANOVA and post-hoc Duncans analysis; i.e. NS=no significant differences at Harper Adams in 2013 or 2014 ($P > 0.05$)

---- Horizontal lines show where the results fall within the Soil index classification system (Defra, 2010) and demonstrate that the soils at Harper Adams only required a maintenance application at Harper Adams to balance K offtake in the crop (Index 2).

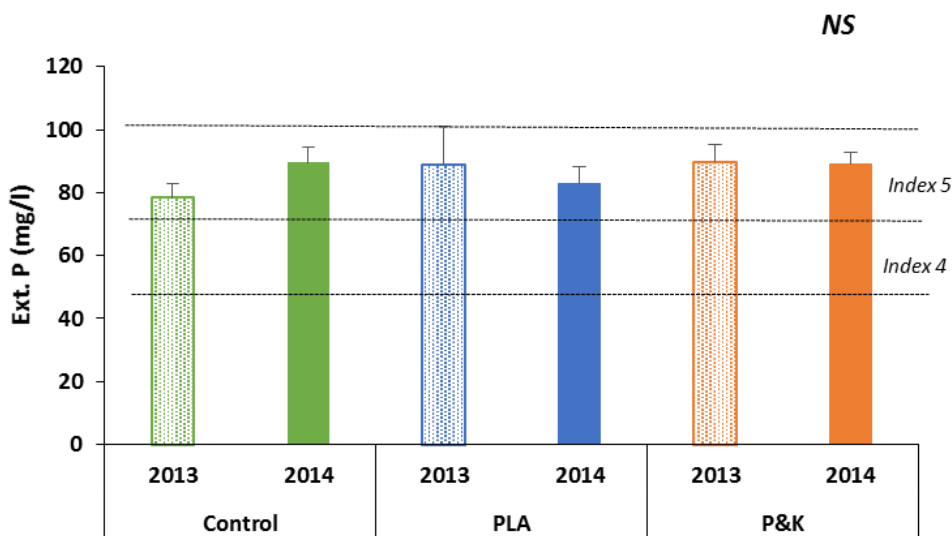
Figure 0-6. Topsoil potassium concentrations measured in April 2013 and 2014: Harper Adams



^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P<0.05$; i.e. at Gleadthorpe in 2014, the PLA^b was significantly different to the control^a and the P&K treatment; NS=no significant in 2013 ($P>0.05$)

---- Horizontal lines show where the results fall within the Soil index classification system (Defra, 2010) and demonstrate that the soils at Gleadthorpe were well-supplied with P

Figure 0-7. Topsoil phosphate concentrations measured in April 2013 and 2014: Gleadthorpe



^{a,b} Different letters between columns indicate significant differences between treatments for a particular year and site at $P<0.05$; i.e. NS=no significant difference at Harper Adams in 2013 or 2014 ($P>0.05$)

---- Horizontal lines show where the results fall within the Soil index classification system (Defra, 2010) and demonstrate that the soils at Harper Adams were well-supplied with P

Figure 0-8. Topsoil phosphate concentrations measured in April 2013 and 2014: Harper Adams

Extractable P concentrations were unaffected by the PLA and P&K applications in 2013 at both sites, but by 2014, concentrations at Gleadthorpe were higher on the PLA treatment compared to the control and equivalent rate of P&K ($P < 0.01$; Figure 0-7 and Figure 0-8), which is most likely a consequence of the higher P loadings to this treatment. Unlike potassium, phosphorus is relatively immobile in soils and is predominantly lost by runoff and erosion, which would have been low as both sites had no gradient. Repeated applications of P fertiliser in excess of crop requirement will therefore tend to result in a build-up of soil P (Johnston & Dawson, 2005).

On all sampling occasions, topsoil extractable P concentrations were well in excess of target indices (at 45-60 mg/l; Index 3-4). There was no build-up of soil P at Harper Adams (Figure 0-8), where concentrations remained at approximately the same level as the previous year, with the soil well-supplied with P (c.86mg/l; Index 5).

The PLA additions, and equivalent P&K treatment, also had no effect on other measured topsoil properties, including the soil microbial biomass and topsoil aggregate stability ($P > 0.05$; Appendix 4).

3.4. Grain yields and uptake of chemicals of potential concern

There were no significant differences in crop yields between any of the treatments ($P > 0.05$) at either site in 2013 or 2014, indicating that all treatments (including the control) were equally beneficial and that the additional P and K supplied by the PLA and P&K treatments were surplus to requirement (Table 0-4). There was also no effect of the PLA and P&K applications on grain nutrient offtake or uptake of chemicals of potential concern (COPCs; Appendix 6).

Table 0-4. Grain yields (t/ha @85% dm) at harvest 2013 (treatment mean with standard error in parenthesis)

Treatment plots	Gleadthorpe		Harper Adams	
	Spring barley (2013)	Winter wheat (2014)	Winter wheat (2013)	Spring barley (2014)
Control	5.1 (0.15)	5.4 (0.36)	4.3 (0.25)	3.1 (0.17)
PLA	5.0 (0.08)	6.2 (0.31)	4.3 (0.22)	2.6 (0.11)
P&K	5.0 (0.21)	6.3 (0.23)	4.3 (0.25)	2.5 (0.40)
P^1	NS (0.62)	NS (0.15)	NS (1.0)	NS (0.33)

¹ Statistical analysis undertaken using ANOVA (data normally distributed); numbers in brackets indicate the P statistic; NS=not significant ($P > 0.05$)

3.5. Storage study

Over the course of the c.12 month storage period there was very little change in the total weight of PLA within the storage heaps which declined by c.5% from 4.8 (± 0.16) tonnes to 4.6 (± 0.06) tonnes (fresh weight). The temperature of the heaps also tended to reflect ambient air temperatures (Figure 0-9), suggesting there were no significant chemical reactions occurring within them. The marginally higher heap temperatures at the outset of the storage period in August 2013 and again at the end of storage in August 2014 was most likely residual heat following summer heating of the PLA (the temperature loggers were buried within the heaps).

Table 0-5. Changes in PLA composition with storage (mean of 3 replicate heaps)

Determinand	Units	Aug. 2013	Sept. 2013	Nov. 2013	Feb. 2014	Sept. 2014	ANOVA ¹		Regression ²	
							P	P	r ²	↕
Dry matter	%	90.3	89.8	80.4	76.0	76.5	<0.001	<0.001	61%	↓
pH	unit	12.2	12.6	12.8	12.4	12.6	NS (0.72)	NS		
Conductivity	µS/cm	90067	91933	106267	nd	61733	<0.001	0.002	61%	↓
Phosphate (P ₂ O ₅)	kg/t fw	171	180	147	146	166	NS (0.24)	NS		
Potash (K ₂ O)	kg/t fw	168	173	165	132	146	<0.001	0.01	36%	↓
Magnesium (MgO)	kg/t fw	49.9	55.1	43.1	47.8	49.4	0.008	NS		
Calcium	kg/t fw	121	122	101	107	110	0.017	NS		
Aluminium	mg/kg dm	3263	3310	2860	3857	3457	<0.001	NS		
Antimony	mg/kg dm	2.80	2.66	3.07	3.72	4.17	<0.001	<0.001	81%	↑
Arsenic	mg/kg dm	10.6	11.3	15.6	7.2	9.66	<0.001	NS		
Barium	mg/kg dm	150	167	155	138	222	<0.001	0.003	47%	↑
Beryllium	mg/kg dm	0.41	0.33	<2	0.45	0.34	<0.001	NS		
Boron	mg/kg dm	125	127	145	102	97.2	<0.001	0.002	49%	↓
Cadmium	mg/kg dm	1.12	1.09	1.24	1.52	1.25	0.002	NS		
Chromium	mg/kg dm	22.4	22.5	20.0	24.8	21.2	<0.001	NS		
Chromium VI	mg/kg dm	<0.3	<0.6	<0.6	<0.6	<0.6	N/A	N/A		
Cobalt	mg/kg dm	4.93	4.87	4.40	5.43	4.84	<0.001	NS		
Copper	mg/kg dm	496	576	427	607	461	<0.001	NS		
Iron	mg/kg dm	4737	4507	3887	5820	4797	0.002	NS		
Lead	mg/kg dm	111	134	130	154	145	0.04	0.04	24%	↑
Lithium	mg/kg dm	6.4	7.9	7.4	10.8	7.84	<0.001	NS		
Manganese	mg/kg dm	2967	2880	2403	3600	3167	<0.001	NS		
Mercury	mg/kg dm	<0.2	<0.2	<0.2	<0.2	<1	N/A	N/A		
Molybdenum	mg/kg dm	32.8	32.3	56.2	6.6	7.03	<0.001	0.01	36%	↓
Nickel	mg/kg dm	15.3	15.0	13.1	18.3	15.2	0.02	NS		
Selenium	mg/kg dm	4.61	4.78	7.14	1.66	2.18	<0.001	0.02	33%	↓
Silver	mg/kg dm	<1	<1	<1	<1	<1	N/A	N/A		
Sodium	mg/kg dm	28900	30633	37067	18467	31000	<0.001	NS		
Strontium	mg/kg dm	231	224	190	253	232	<0.001	NS		
Thallium	mg/kg dm	1.91	1.43	<1	<1	1.01	N/A	N/A		
Tin	mg/kg dm	2.96	3.30	3.34	4.07	3.73	0.01	0.02	28%	↑
Titanium	mg/kg dm	335	354	339	340	359	NS (0.75)	NS		
Vanadium	mg/kg dm	16.6	16.5	20.7	11.9	13.2	<0.001	0.04	23%	↓
Zinc	mg/kg dm	2103	2160	2040	1647	2390	<0.001	NS		

¹Statistical analysis undertaken using ANOVA (data normally distributed); NS=not significant ($P>0.05$); N/A=not applicable (results below the limit of detection)

²Statistical analysis undertaken using Regression; NS=not significant ($P>0.05$), r^2 = percentage of variance accounted for; ↕=direction of change (↑= increase; ↓decrease)

As the heaps were uncovered, the moisture content increased (from c.10% to 25%) and total dry matter decreased ($P < 0.001$; Table 0-5). There was considerable variation in PLA composition over time ($P < 0.001$; by ANOVA) for most of the measured parameters (Table 0-5). However, the only consistent variation (as determined by regression analysis) was for total potash, which declined by c.15% ($r^2 = 36\%$) and conductivity, which declined by c.30% ($r^2 = 61\%$).

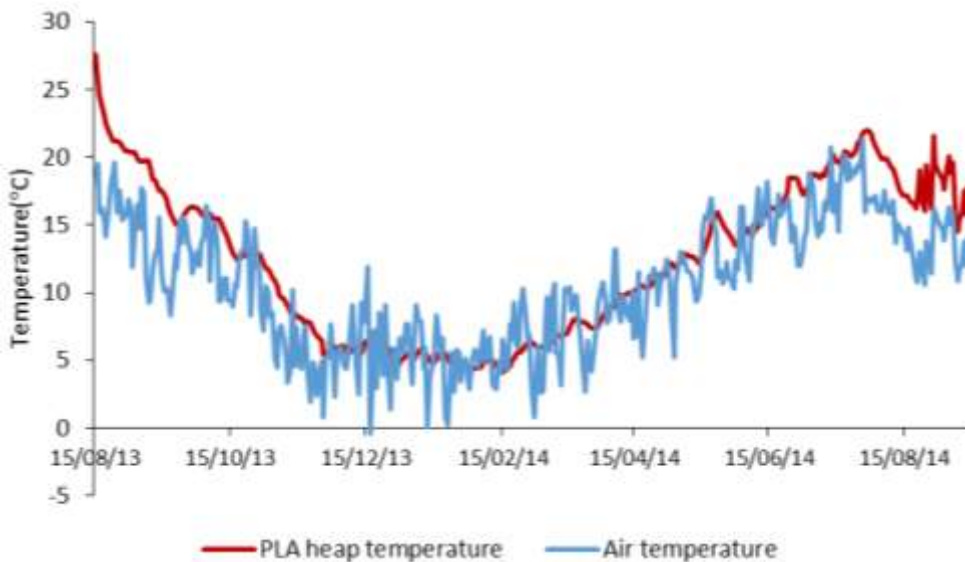


Figure 0-9. Average daily air and heap temperatures

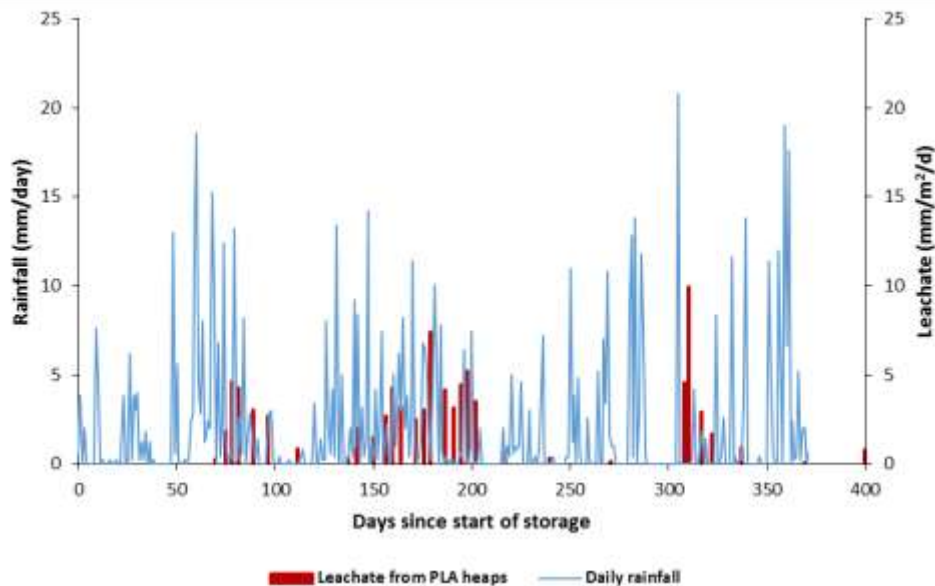


Figure 0-10. Leachate loss patterns and total rainfall during storage of the PLA

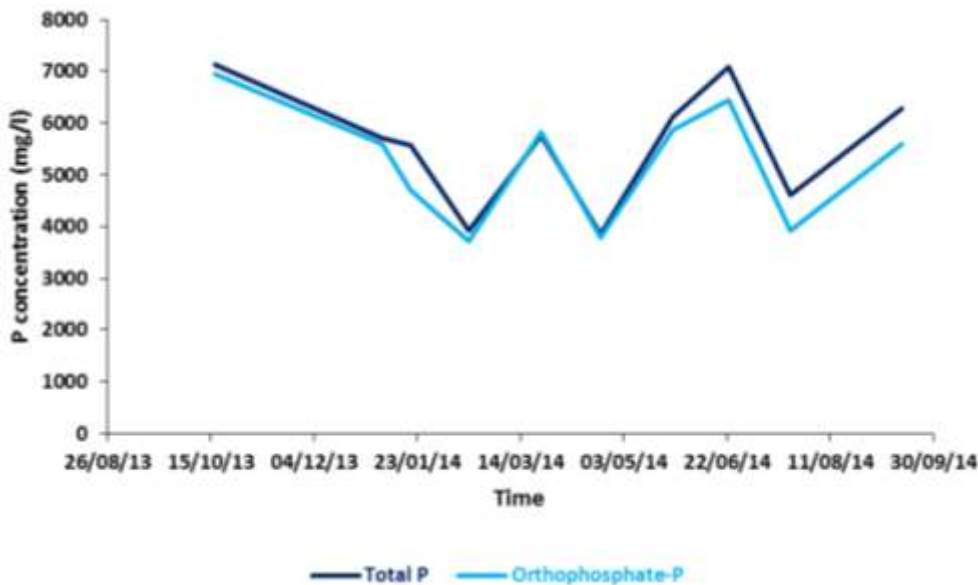


Figure 0-11. Phosphorus concentrations in water draining from the PLA heaps

Almost all the total P draining from the PLA heaps was 'reactive', i.e. orthophosphate P (Figure 0-11). Standards for the concentrations of molybdate reactive P (MRP) in rivers were recently updated as part of the UK's implementation of the Water Framework Directive (Anon, 2014). Here, lowland rivers with a MRP concentration of <math><0.11\text{ mg/l}</math> are considered to be of moderate or good quality, whilst concentrations >math>0.8\text{mg/l}</math> are considered to be poor quality (Anon, 2014).

Concentrations of orthophosphate P in the drainage from the PLA heaps were well in excess of these levels (4000-7000 mg/l), with no significant change in concentration for the duration of the study ($P>0.05$; Table 0-6), with losses amounting to c.25 kg total P (equivalent to c.7% of the P stored in the heaps, Table 0-7). This is not surprising, given PLA is predominately an inorganic P_2O_5 and K_2O fertiliser and as such should not be stored outside for extended periods (note: K concentrations were not part of the suite of analyses performed on the leachates).

The pH of the leachate averaged 12.5 for the duration of the study, albeit with a slight decline over the c.12 month period (with regression analysis only accounting for 16% of the variation in pH, Table 0-7). At these pH levels, the leachate was highly reactive and in considerable excess of the Fresh Water Fish Directive Imperative values of pH 6-9 for Salmonid and Cyprinid waters (EU, 2006).

There was considerable variation in the concentrations of other nutrients and metals within the leachate ($P<0.05$; by ANOVA; Table 0-5). The concentration is a function of both the quantity leached and the volume of leachate. As can be seen from Table 10, the volume collected in the drainage tanks varied over the year, reflecting rainfall patterns (Figure 0-10), with more rainfall over the winter months, and only small volumes collected in July. This could lead to more diluted leachate over winter and increasing concentrations in the summer. However, most of the measured parameters declined in concentration over time (Table 0-6).

For the majority of metals, the total amount leached only represented a small proportion of that stored in the heap (<math><1\%</math>; Table 0-7). However, for arsenic, vanadium, selenium and molybdenum the proportion lost was much greater (Table 0-7), with almost all the molybdenum (c.99%) stored within the PLA lost via leaching at the end of the storage period. As PLA is marketed not only for its P and K supply properties, but also as a source of trace elements (including molybdenum and selenium), this represents a loss of valuable nutrients.

Table 0-6. Composition of the leachate draining from the PLA heaps.

Determinand	Units	Sample date ¹										ANOVA ²		Regression ³	
		17/10/13	06/01/14	20/01/14	17/02/14	24/03/14	22/04/14	27/05/14	23/06/14	23/7/14	15/9/14	P	P	r ²	↕
Volume of water in tank on sample date	litres	104	192	181	144	65	39	186	128	20	232				
Total drainage between samplings	litres	104	1362	442	1219	157	39	186	594	20	232				
pH		13.1	13.2	13.2	13.1	12.4	11.4	12.9	12.7	10.4	12.7	0.005	0.04	17	↓
BOD	mg/l	<14.6	<9.73	<5.84	<2.92	<29.2	<2.92	<29.2	<2.92	89.7	<2.92	N/A			
Total P	mg/l	7130	5700	5575	3915	5745	3855	6120	7075	4620	6290	NS (0.33)	NS		
Orthophosphate-P	mg/l	6937	5595	4695	3705	5815	3780	5870	6435	3925	5590	NS (0.37)	NS		
Total N	mg/l	95.8	73.4	55.4	20.8	25.6	31.25	24.6	22.8	54.2	13.9	0.02	0.003	35	↓
Ammonium N	mg/l	<0.5	<0.5	<0.5	<0.5	<0.5	1.36	<0.5	<0.5	8.06	<0.5	N/A			
Total oxidised N	mg/l	1.2	<1	<1	<1	<1	1.1	<1	<1	<1	<1	N/A			
Aluminium	ug/l	2917	1710	1385	463	1076	150	657	659	910	113	NS (0.12)	0.03	19	↓
Antimony	ug/l	110	18.3	79.9	55.3	67	51	67	131	42	94	0.01	NS		
Arsenic	ug/l	12417	6880	5785	3990	6015	4330	5470	5010	2505	3170	NS (0.1)	<0.001	44	↓
Barium	ug/l	39.5	<300	<30	<30	<30	40	14	<12	81	<60	N/A			
Beryllium	ug/l	<200	<100	<10	<4	<10	<10	<2	<20	<4	<20	N/A			
Cadmium	ug/l	15.9	12.4	12.05	1.39	3.0	1.0	1.0	1.0	4	7	<0.001	0.004	35	↓
Chromium	ug/l	688	226	212	135	131	25	135	211	175	275	NS (0.18)	NS		
Chromium VI	ug/l	<3	<3	<3	<3	<3	<3	<3	<3	0	0	N/A			
Cobalt	ug/l	29.4	26.6	21.9	5.69	11	6.0	9.0	7.0	6.93	4.90	<0.001	<0.001	58	↓
Copper	ug/l	701	206	117	1730	552	2095	702	1955	862	892	<0.001	NS		
Lead	ug/l	72.5	122	96.6	2.53	56	<2	<100	11	54	3.91	NS (0.24)	0.03	26	↓
Manganese	ug/l	137	104	94.9	<4	95	8.0	49	9.0	212	18	<0.001	NS		
Mercury	ug/l	<0.2	0.10	<0.02	0.16	0.19	0.36	0.24	0.21	0.34	0.21	0.014	0.05	16	↑
Molybdenum	ug/l	71600	46350	37250	8745	8905	5715	5145	5295	2630	3625	0.001	<0.001	63	↓
Nickel	ug/l	103	122	97	48	68	45	57	64	30	47	0.04	0.003	36	↓

Selenium	ug/l	2063	1097	945	579	228	385	128	313	122	230	0.01	<0.001	61	↓
Silver	ug/l	<60	<200	<20	<20	<20	<17.5	<3.5	<7	<7	<40	N/A			
Tellurium	ug/l	0.86	<20	<2	<2	<2	<20	<0.4	<0.8	<0.8	<4	N/A			
Thallium	ug/l	68.9	<200	<20	<8	<20	<20	<4	<8	<20	<80	N/A			
Tin	ug/l	10.3	<200	<20	<20	<20	<20	<4	8.0	<8	<40	N/A			
Titanium	ug/l	359	307	489	126	385	192	432	385	296	570	0.002	NS		
Uranium	ug/l	4.39	11.22	9.68	2.06	14	3.0	17	4.0	<2.5	6	NS (0.41)	NS		
Vanadium	ug/l	12933	9550	8020	3500	5045	2750	4095	4075	1490	3010	0.006	<0.001	58	↓
Zinc	ug/l	754	1800	923	<60	588	<60	294	<300	604	<300	N/A			

¹mean of 3 replicate heaps in October and 2 replicate heaps thereafter;²Statistical analysis undertaken using ANOVA (data normally distributed); Numbers in brackets indicated the P statistic; NS=not significant ($P>0.05$); N/A=not applicable (results below the limit of detection);³Statistical analysis undertaken using Regression; NS=not significant ($P>0.05$), r^2 = percentage of variance accounted for; ⚡=direction of change (↑= increase; ↓decrease)

Table 0-7. Total losses in relation to heap composition (mass balance)

Determinand	Total in heap (kg)		Change in heap		Total leached		
	Aug.13	Sept. 14	kg ¹	% of initial heap content	kg	% of initial heap content	% of change in heap
Total P	360	332	28.1	7.80	24.9	6.92	88.7
Aluminium	14.2	12.1	2.10	14.8	0.006	0.04	0.29
Antimony	0.01	0.02	-0.002	-20.1	3.02E-04	2.48	-12.4
Arsenic	0.05	0.03	0.012	26.4	0.029	63.0	239
Cadmium	0.01	0.01	0.001	10.4	3.73E-05	0.77	7.34
Chromium	0.10	0.07	0.023	24.1	0.001	1.27	5.28
Cobalt	0.02	0.02	0.005	21.1	7.80E-05	0.36	1.72
Copper	2.16	1.61	0.545	25.3	0.003	0.16	0.62
Lead	0.48	0.51	-0.024	-5	2.84E-04	0.06	-1.16
Manganese	12.91	11.1	1.821	14.1	3.42E-04	0.00	0.02
Molybdenum	0.141	0.03	0.118	82.7	0.141	98.7	119
Nickel	0.07	0.05	0.013	20.2	3.67E-04	0.55	2.72
Selenium	0.02	0.01	0.012	61.9	0.004	20.2	32.6
Titanium	1.46	1.26	0.202	13.9	0.002	0.10	0.74
Vanadium	0.07	0.05	0.026	36.0	0.032	44.3	123
Zinc	9.15	8.37	0.784	8.6	0.003	0.04	0.42

¹Changes in bold are significant at $P < 0.05$ by regression (see Table 9); mass balance only calculated for those elements where concentrations in the leachate were greater than the analytical limits of detection.

The PLA Quality Protocol states that producers, distributors and users should follow good practice for the storage of PLA, treating it as an agricultural fertiliser. For inorganic fertilisers, this means storing the material undercover in a cool, dry shed. Indeed, the safety data sheet provided by the PLA industry recommends storage in a cool, dry, well-ventilated place away from direct sunlight. It also states that as a strong alkaline (pH > 12 in solution) it can react with strong acids and metals when exposed to moisture as well as react with ammonium salts to release ammonia gas.

Therefore, PLA should not be stored in field heaps in normal operational practice and following on-farm deliveries, spreading almost always occurs within a day(s) to avoid compression of the product before spreading.

3.6. PLA Heap 3

In November 2013 (c.3 months after establishment), a reduction in leachate volume was noticed from PLA replicate Heap 3. Upon further investigation, a leak was identified, emanating from the pipework exiting the hydrologically isolated cell. Attempts to fix the problem revealed salt crystals in the pipework. Although these were removed, the leak remained and it was not possible to collect leachate from this heap for the remainder of the storage period. As the other two PLA heaps were functioning normally this did not present a problem for the validity of the storage study.

The heaps were established in sloping concrete cells lined with plastic, with a short length of perforated plastic drainage pipe placed at the bottom of the slope to collect leachate and direct it into a solid pipe exiting from the lowest corner of the cell and into a collection tank.

Further investigation at the end of the storage period after removal of the PLA from the cell, revealed salt crystals along the length of the perforated plastic pipe (similar to those collected earlier in the year from the solid pipework exiting the cell). The crystals were copper in colour (Figure 0-12) – a characteristic associated with potash crystals (i.e. potassium and sodium chloride). This was confirmed by subsequent analysis of the crystals, which contained over 40% potassium (Table 0-8).



Figure 0-12. Salt crystals on the pipework within PLA Heap 3

Table 0-8. Chemical composition of the crystals within the pipework of PLA Heap 3

Determinand	Concentration (g/kg)
Potassium	434
Sodium	4.22
Calcium	2.40
Magnesium	0.70
Phosphorus	1.98
Chloride	3.04

It is believed therefore that the reduction in leachate volume and subsequent leak from PLA Heap 3 was due to the crystallization of potassium salts which blocked the perforations in the pipework at the base of the heap and prevented water from reaching the solid exit pipe. It is unclear why this was only a problem for PLA Heap 3 and not the other two replicated heaps. As PLA should not be stored outside (for extended periods), this crystallisation should not pose a problem during normal operational use of PLA if good practice is followed.

Discussion of research questions

1. *How do physical and chemical properties of soil and pore water change over time?*

There was very little change in soil properties on the control and P&K treatments over the course of the three sampling events (i.e. site characterisation in January 2013, April 2013 and April 2014). However, where PLA had been applied at Gleadthorpe, topsoil extractable P concentrations increased over time (Figure 0-7) from 41 mg/l (Index 3) at site characterisation in January 2013 to 60 mg/l (Index 4) in April 2014, following two annual applications of PLA ($P < 0.05$). This confirmed its value as a P fertiliser useful for building up soil P levels on low Index soils.

This effect was not apparent on the P&K treatment (the standard alternative to PLA) at Gleadthorpe. It was thought likely that this was due to the higher P loading rates from the PLA, with TSP application rates based on the *standard* PLA compositional analysis (as supplied with the PLA), rather than the *actual* compositional analysis post-spreading (which indicated a higher P content). There was also no significant change in topsoil P concentrations at Harper Adams (Figure 0-8), most likely because the soils here were well-supplied with P_2O_5 (c.86mg/l; Index 5).

PLA applications also caused a temporary increase in topsoil sodium (at both sites) c.2 months after application in April 2013 (Figure 0-1 and Figure 0-2) which had disappeared by April 2014 (c.6 months after the second annual application). This was most likely due to dissolution and equilibration into the topsoil, as well as over-winter leaching. Topsoil strontium was also temporarily elevated at Harper Adams in April 2013 (Figure 0-4), most likely due to the presence of PLA material within the soil sample as the material had been top-dressed onto the growing winter wheat crop, whereas PLA applications prior to the 2014 sampling were incorporated into the soil c.6 months prior to soil sampling.

This pattern of elevated concentrations shortly after application, in April 2013, falling to background concentrations in April 2014, was also apparent for total and extractable potassium, total beryllium, iron and titanium at Gleadthorpe and pH at Harper Adams ($P < 0.05$ for change over time), although differences between treatments for these parameters were inconsistent.

Insufficient pore water could be extracted from the soils for any meaningful analyses to be performed due to the inherently low water retention on these sandy soils.

2. *How do physical and chemical properties of soil and pore water in plots with application of PLA compare with those in plots with industry standard alternatives/equivalents and control plots?*

Two applications of PLA resulted in an increase in topsoil extractable P concentrations at Gleadthorpe relative to the control and P&K treatment (Figure 0-7). This was not apparent at Harper Adams, where soils were already well-supplied with P (Figure 0-8).

There was no significant effect of PLA additions on topsoil extractable K concentrations at either site in 2013 or 2014 ($P > 0.05$; Figure 0-5 and Figure 0-6). At Gleadthorpe, extractable K ranged between 60-100 mg/l (Index 1) suggesting additional K was required to maximise yields. Concentrations were higher at Harper Adams where extractable K averaged 168 mg/l and 150 mg/l in 2013 and 2014, respectively (Index 2-), with no difference between treatments. The higher K loading on the PLA treatment had no impact on topsoil extractable or total K levels. These results suggest that all the K supplied with the treatments (both the PLA and MOP) was either utilised by the crop (particularly at Gleadthorpe where the soils were low in extractable K) or was leached.

Sandy soils, such as those at Gleadthorpe and Harper Adams, typically have a low cation exchange capacity and are therefore less likely to retain mobile cations like potassium. Consequently, they often require annual potash applications to maintain crop yields and soil K status.

PLA applications resulted in a temporary increase in topsoil sodium and strontium concentrations ($P < 0.05$) shortly after the first application in 2013. Although higher than the control and P&K treatment, concentrations of both elements were still at the low end of the range of reported values for soils in England and Wales, which average 79 mg/kg (range: 4.5- >1000 mg/kg) for strontium and 0.32% or 3200 mg/kg (range: 0.074-7.4%) for sodium (Rawlins *et al.*, 2012).

Concentrations declined over the subsequent 12 months such that by April 2014 there was no significant difference between the treatments, despite a second application of PLA in Autumn 2013. It is thought this initial increase is due to the presence of PLA within the soil sample (which was taken c.2 months after application and incorporation in Spring 2013, and at Harper Adams the PLA was top-dressed onto the growing crop, without soil incorporation).

The PLA and equivalent P&K additions also had no significant effect soil aggregate stability in 2013 or 2014.

3. To what extent are COPCs taken up by crops grown with application of PLA?

The application of PLA caused no significant additional uptake of COPCs relative to the untreated control or the conventional P&K treatment over the timescales of the study.

4. How does uptake of COPCs by crops grown with application of PLA compare to uptake by crops grown with application of industry standard alternatives/equivalents and in control plots?

The two annual applications of PLA caused no significant additional uptake of COPCs relative to the untreated control or the conventional P&K treatment over the timescales of the study.

5. How does the total soil microbial biomass change over time in soils with application of PLA?

There was no significant change in soil microbial biomass over time where PLA had been applied. At Gleadthorpe, microbial biomass C averaged 33 mg/kg between 2013 and 2014 (range 32-34 mg/kg) where PLA had been applied, whereas at Harper Adams the microbial biomass pool was higher and averaged 218 mg/kg (range 199-238 mg/kg).

6. How does total soil microbial biomass in soils with application of PLA compare with total soil microbial biomass with application of industry standard alternatives/equivalents and in control plots?

There was no significant difference in microbial biomass between the PLA treatment, industry standard P&K treatment or untreated control in either 2013 or 2014 ($P > 0.05$). The absence of any treatment effects at both sites suggests the applied PLA did not have a significant effect on soil biological functioning.

7. How do storage durations affect key properties of the PLA (e.g. nutrient content, pH, pathogens)?

The storage study demonstrated that unprotected storage outside in field heaps results in a decline in the fertiliser value of the material (i.e. a loss of both major and trace nutrients, specifically potassium, boron, molybdenum and selenium); and leads to the generation of a highly reactive alkaline leachate containing elevated concentrations of soluble P which would be highly detrimental if it entered surface water bodies in an un-diluted form.

The PLA Quality Protocol (QP) states that producers, distributors and users should follow good practice for the storage of PLA, treating it as an agricultural fertiliser. For inorganic fertilisers, this means storing the material undercover in a cool, dry shed. The safety data sheet provided by the PLA industry recommends storage in a cool, dry, well-ventilated place away from direct sunlight. PLA should therefore not be stored in field heaps in normal operational practice and, following on-farm deliveries, spreading should occur within days to avoid compression of the product before spreading.

The results of the storage study highlights the importance of the QP recommendations, i.e. that PLA should not be stored outside in field heaps.

8. How do the yields of crops grown with application of PLA compare with those grown with application of industry standard alternatives/equivalents and in control plots?

There was no significant difference ($P > 0.05$) in crop yields between the PLA, industry standard P&K fertilisers and the untreated control at either of the sites over two cropping seasons (Table 0-4). Both sites had adequate soil P reserves at the outset of the trials (January 2013), with extractable P concentrations ranging from 41-54 mg/l at Gleadthorpe (Index 3/4) and from 80-101 mg/l at Harper Adams (Index 5).

The baseline extractable K concentrations at Gleadthorpe (63-69 mg/l; Index 1) suggested additional fertiliser K would be required, whereas at Harper Adams, the higher concentrations (164-185 mg/l; Index 2) suggested only a maintenance dressing was necessary (to balance crop offtake). As all treatments received recommended rates of N & K fertiliser as required (following the 'Fertiliser Manual – RB209; Defra, 2010), this clearly balanced the nutrient supply across all treatments resulting in similar yields.

In order to evaluate the fertiliser value of PLA, experiments should be conducted on lower index soils, and include a range of inorganic P and K fertiliser rates as treatments (to generate a yield response relationship) compared to PLA applied at recommended rates (with no additional inorganic fertiliser).

The results from the field trial demonstrated that two years of PLA applications had no significant detrimental effects on crop yields relative to the industry standard and control.

9. How do the yield benefit-costs of PLA compare to those of industry standard alternatives/equivalents?

PLA is typically c.20% cheaper than the equivalent amount of Triple Super Phosphate-TSP and Muriate of Potash -MOP¹). As there was no significant difference in the yields between treatments, this would suggest PLA may be economical compared to the industry equivalents. However, given both soils were adequately supplied with P, there was no crop requirement for additional P fertiliser.

Likewise, all treatments received recommended rates of K fertiliser (including the PLA treatment). It is therefore not possible to assess robustly the yield benefit-cost for PLA from the field trials, further work would be needed. In order to evaluate fully the economic benefit of PLA, a future bespoke study on lower index soils would be recommended.

¹ www.nutrifocus.co.uk

Conclusions

This study evaluated the impact of PLA relative to an industry standard equivalent (i.e. inorganic P and K fertiliser) on selected chemical, physical and biological properties of sandy soils growing cereals in a temperate climate over a two year period.

The results demonstrated that PLA is beneficial as a fertiliser and indicated that PLA can be used for building up soil P levels on low Index soils.

The results showed no significant negative effect on a range of soil chemical, physical and biological properties, or on the uptake of COPCs by cereal crops, over the timescales of the study. This clearly demonstrated that the two applications of PLA to agricultural soils presented no significant environmental or human health risks.

However, the storage study reinforced the need to store PLA in a dry environment, in accordance with the Quality Protocol for PLA, in order to prevent the generation of potentially harmful leachate.

The field trials have improved understanding of the environmental impacts and agricultural benefits of the application of PLA to agricultural soils. The study also demonstrated the importance of adhering to the Quality Protocol with respect to storing PLA materials in temporary field heaps to prevent surface water pollution.

The field trials have provided useful data and understanding to inform future QRAs. The objectives for the EQual PLA field trials have therefore been achieved.

It would be beneficial to undertake a longer term study to assess whether COPCs build up through long-term use. It may also be beneficial to carry out further research on low P and K index soils, to provide additional information with regard to the magnitude of economic benefits of PLA applications compared to non-waste derived alternatives.

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References

- ANON, 1982. Techniques for Measuring Soil Physical Properties. MAFF Reference Book 441. HMSO, London.
- ANON, 1986. The Analysis of Agricultural Materials. MAFF Reference Book 427. HMSO, London.
- ANON, 2014. Water Framework Directive implementation in England and Wales: new and updated standards to protect the water environment. Crown Copyright, May 2014. www.gov.uk/defra.
- BHO GAL, A., NICHOLSON, F.A. AND CHAMBERS, B.J. (2009). Organic carbon additions: effects on soil biophysical and physico-chemical properties. *European Journal of Soil Science*, **60**, 276-286.
- DEFRA, 2010. The Fertiliser Manual (RB209). The Stationery Office, Norwich
- EA. 2012. EQual programme - Market survey for paper sludge spread to land. Final Report, July 2012. Environment Agency, Bristol.
- EA AND WRAP. 2012. Poultry Litter Ash. End of waste criteria for the production and use of treated ash from the incineration of poultry litter, feathers and straw. WRAP, Banbury.
- EU. 2006. Directive 2006/44/EC of the European Parliament and of the Council of 6 September 2006 on the quality of fresh waters needing protection or improvement in order to support fish life. <http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri-OJ:L:2006:264:0020:0031:EN:pdf>.
- GIBBS, P., MUIR, I, RICHARDSON, S., HICKMAN, G. AND CHAMBERS, B., 2005. Landspreading on agricultural land: nature and impact of paper wastes applied in England and Wales. Science Report SC030181/SR, Environment Agency, Bristol.
- JENKINSON, D.S. AND POWLSON, D.S., 1976. The effect of biocidal treatments on metabolism in soil. V. A method for measuring soil biomass. *Soil Biology and Biochemistry*, **8**, 209-213.
- JOHNSTON, A.E. AND DAWSON, C.J. 2005. Phosphorus in Agriculture in Relation to Water Quality. Agriculture Industries Confederation. November 2005; Peterborough.
- MAFF (1983) Lime and Liming. MAFF Bulletin 35, HMSO London.
- RAWLINS, B.G., McGRATH, S.P., SCHEIB, A.J., BREWARD, N., CAVE, M., LISTER, T.R., INGHAM, M., GOWING, C. AND CARTER, S. 2012. The advanced soil geochemical atlas of England and Wales. British Geological Survey, Keyworth. www.bgs.ac.uk/gbase/advsoilatlasEW.html.

List of abbreviations

ANOVA	Analysis of Variance
BOD	Biological Oxygen Demand
COPC	Chemicals of Potential Concern
GT	Gleadthorpe field trials site
HA	Harper Adams field trials site
MOP	Muriate of Potash
PLA	Poultry Litter Ash
PS	Paper Sludge
TSP	Triple Super Phosphate

Appendix 1: Field Study Method Statements

EQual Project (30636) Method Statement:

Baseline Soil Sampling & Analysis

This method statement details the procedures to be followed in soil sampling the two EQual field trials sites (ADAS Gleadthorpe and Harper Adams Agricultural College) at the outset of the study.

In order to characterise the site, 'baseline' topsoil samples (0-15cm) should be taken from each plot at Gleadthorpe prior to any treatment applications. At Harper Adams, samples should only be taken from the untreated control, poultry litter ash (PLA) and fertiliser treatments (i.e. excluding the Paper Sludge (PS) treatment).

Equipment

- Soil sampling for the chemical analyses will be taken using a hand-held "Cheese" corer. This is suitable for most soil conditions.
- A spade or trowel for aggregate stability sampling.
- Soil spatula, for use with cheese corer.
- Ruler graduated in cm.
- New clean polythene bags 300 x 200 mm to 500 x 400 mm, labelled accordingly
- Rubber bands or ties (or use self-seal bags)
- Boxes for storage of samples.
- 1kg plastic tubs (supplied by NLS)
- 1 litre glass jars.
- Field trials site plan.

Sampling Procedures

The following samples should be taken when the soil is moist, but not frozen or too wet:

- Soil samples from 0-15cm depth will be taken following the methodology detailed in the "Fertiliser Manual (RB209)" (Defra, 2010; Appendix 3). Twenty soil core samples will be taken at even intervals in a 'W' shape pattern across each plot, using a hand held "Cheese corer", giving the specified volume of soil. The soil from each plot will be collected in one bag to generate one composite (representative) sample from each plot. Samples should be placed in 1 kg plastic tubs (3 per plot) and sent fresh in cool boxes to the NLS laboratory (store in a fridge at <4°C prior to dispatch).
- A further c.1kg of topsoil (0-15cm) will be collected according to the methodology detailed in the "Fertiliser Manual (RB209)" (Defra, 2010) for the determination of soil microbial biomass C & N. Twenty soil core samples will be taken at even intervals in a 'W' shape pattern across each plot, using a hand held "Cheese corer". The soil from each plot will be collected in one bag to generate one composite (representative) sample from each plot. Samples should be placed in plastic bags and sent fresh in cool boxes to the ADAS laboratory at Boxworth (F.A.O. Helen Kingston/Masuma Chauhan). Store in a fridge at <4°C prior to dispatch.
- Using a trowel/spade, twenty topsoil (0-15cm) samples will be taken from each plot spread evenly in a 'W' shape pattern to provide c.1.0kg of bulked fresh soil sample per plot. The soil sample should be sent to the ADAS laboratory at Boxworth (F.A.O. Helen Kingston/Masuma Chauhan) for the determination of aggregate stability. Care should be taken when sampling and during transit of these samples, in order to minimise disruption of the soil aggregates. Samples should be sent to the laboratory in boxes, and should not be stacked on top of each other.

- A further c.1kg topsoil (0-15cm) will be taken following the methodology detailed in the “Fertiliser Manual (RB209)” (Defra, 2010) for storage in the sample archive at ADAS Gleadthorpe. Samples should be stored in 1 litre glass jars.

Labelling

All samples will be labelled using the following method:

EQual YAS2002/SITE/PLOT NUMBER/DATE

Samples will not be dispatched on a Friday, as they may deteriorate over the weekend. Samples will be stored in a fridge (<4°C) prior to transit.

Roles and Responsibilities

Sampling is to be conducted by a team led by the Site Manager

Data Recording

An entry will be made in the experimental diary to record the date and time of soil sampling, weather and ground conditions, plots that were sampled, sampler name and laboratory dispatch date and details.

Health and Safety

- There is a risk of contracting tetanus from soil. Anyone who takes soil samples should ensure they maintain a course of injections to protect themselves against tetanus infection.
- Efficient soil sampling tools have sharp edges; care should be taken when they are used.
- Back injuries can occur when using soil sampling tools. This risk can be minimised by using a good lifting technique, i.e. keep the back straight and bend the knees.
- The site specific risk assessment at each experimental location (which already considers soil sampling) will be consulted before and after sampling. If any amendments are required, the risk assessment will be updated.

References

Defra (2010). The Fertiliser Manual (RB209) 8th edition. The Stationery Office, Norwich

**EQual Project (30636) Method Statement:
Microbial Biomass Carbon and Nitrogen Analysis**

This method statement details the procedures to be followed in determining the microbial biomass carbon (C) and nitrogen (N) content of soil sampled from the two EQual experimental sites (ADAS Gleadthorpe and Harper Adams University) in February 2013 ('baseline'), March/April 2013 ('year 1') and March/April 2014 ('year 2').

Microbial biomass C & N will be determined at ADAS Boxworth using the fumigation-extraction method (Jenkinson & Powlson, 1976; Wu *et al.*, 1990), with extracts sent to NRM laboratories for analysis. Extractions will be performed within 3 months of receipt of the soil samples.

Equipment

- 6.25mm mesh sieve.
- Tissue/Cotton wool.
- Incubator set at 25 °C.
- 250ml plastic screw-top bottles.
- 60/100ml glass jars or beakers.
- Dry matter tins & oven.
- Dessicator.
- Fume cupboard.
- Vacuum pump.
- Reciprocal shaker.
- 30ml sample bottles.
- 15cm Whatman GF/A filter papers.

Reagents

- Soda Lime (1.0-2.5mm, non-hygroscopic granules).
- Ethanol-free Chloroform (CHCl₃).
- 0.5M Potassium sulphate (K₂SO₄).
- Anti-bumping granules (fused alumina).

Analysis Procedure

Biomass C & N should be determined on soils at 50% water holding capacity, WHC (\pm 2%). Therefore, upon receipt of the samples the gravimetric moisture content must be determined immediately and WHC adjusted accordingly. The method used to determine soil microbial biomass C & N is that of Jenkinson & Powlson (1976) and Wu *et al.* (1990). Chloroform is used to fumigate a soil sample and kill the soil microbial population, resulting in the release of microbial C and N. This is extracted using potassium sulphate solution, with the extracts analysed for total organic C and N (at NRM laboratories). Biomass C & N can then be calculated from the difference in organic C and N extracted from fumigated and unfumigated soil samples. The analysis must be performed within 3 months of receipt of the soil samples, with extracts sent to NRM laboratories within 48 hours of extraction. All samples (soils and extracts) will be stored in a fridge (< 4 °C) prior to analysis. A 1 litre sample of blank K₂SO₄ will be sent to NRM laboratories with each batch of samples for matrix matching.

Labelling

All samples will be labelled using the following method:

EQual YAS2002/SITE/PLOT NUMBER/DATE

Samples will not be dispatched to NRM laboratories on a Friday, as they may deteriorate over the weekend. Samples will be stored in a fridge (<4°C) prior to transit.

Roles and Responsibilities

Extraction is to be conducted by a team led by the Laboratory Manager at ADAS Boxworth.

Data Recording

An entry will be made in the experimental diary to record the date of analysis and analyst, as well as the NRM laboratory dispatch date and details. Data will be recorded electronically (example spreadsheet in Appendix 1).

Health and Safety

- There is a risk of contracting tetanus from soil. Anyone who takes soil samples or working with soil should ensure they maintain a course of injections to protect themselves against tetanus infection. Protective clothing (e.g. goggles, gloves, laboratory clothing) should be worn and hands washed before eating, drinking or smoking.
- Chloroform is classified as a harmful product which can cause serious damage to health by prolonged exposure through inhalation and if swallowed. It is essential that its use is confined to an efficient fume cupboard and that eye protection and gloves are worn when handling this substance.
- Soda lime is 75% Ca(OH)₂ and 3.5% NaOH (the balance is water), which is a caustic irritant if dust is inhaled, or contact made with eyes or broken skin. It is essential that gloves and eye protection are worn when handling this substance.
- A laboratory risk assessment for each analytical procedure is currently in place and will be consulted before and after analysis. If any amendments are required, the risk assessment will be updated.

References

Jenkinson, D.S. & Powlson, D.S. (1976) The effect of biocidal treatments on metabolism in soil. V. A method for measuring soil biomass. *Soil Biology and Biochemistry*, **8**, 209-213.

Wu, J., Joergensen, R.G., Pommerening, B., Chaussod, R. & Brookes, P.C. (1990) Measurement of soil microbial biomass C by fumigation-extraction – an automated procedure. *Soil Biology and Biochemistry* **22**, 1167-1169.

Example excel spreadsheet for the recording and calculation of soil microbial biomass C and N

Filename:

MEASUREMENT OF MICROBIAL BIOMASS

PROJECT CODE:
PROJECT TITLE:
SITE:
DATE SAMPLES TAKEN:
NUMBER OF SAMPLES:

Received at Boxworth: Name: Date: Blank TOC mg/l:
Where stored at Boxworth: Name: Date: Blank TN mg/l:
Soils Processed Name: Date:
Extracts to NRM labs Name: Date:

Correction factors for conversion of EN or EC to BN or BC:

TOC : Shimadzu TOC-VCPH analyser: KC = 0.45 (2.22)
Npers: UV persulphate oxidation: KN = 0.45 (2.22)

Sample ID/Plot	Sample Type	Results from NRM laboratories		net TN in 0.5M K2SO4 mg/l	net TOC mg/l in 0.5M K2SO4	%SMC	DW Soil	Vol. Extract used (ml)	N (mg/kg)	TOC (mg/kg)	Nitrogen (EN) (mg/kg)	Carbon (EC) mg/kg	Biomass N (BN) mg/kg	Biomass C (BC) mg/kg
		TN in 0.5M K2SO4 mg/l	TOC mg/l in 0.5M K2SO4											
	Extraction			0.00	0.0		50	200	0.0	0.0	0.0	0.0	0.0	0.0
	Fumigation													
	Extraction													
	Fumigation													

EQual Project (30636) Method Statement:

Aggregate Stability Analysis

This method statement details the procedures to be followed in determining the aggregate stability of soil sampled from the two EQual experimental sites (ADAS Gleadthorpe and Harper Adams University) in February 2013 ('baseline'), March/April 2013 ('year 1') and March/April 2014 ('year 2').

Soil aggregate stability will be determined at ADAS Boxworth, using the dispersion ratio method of aggregates in the size range 10 – 20mm (Anon, 1982).

Equipment

- Air dried sample of soil aggregates in the size range 10-20mm.
- Plastic measuring cylinders, volume 1000 ml.
- Laboratory film (100 mm wide Nescofilm).
- 20 ml pipette grade B.
- Rubber bands (2 per measuring cylinder).
- Balance with a resolution of 0.01 g or better.
- Balance with a resolution of 0.0001 g or better.
- 50 ml glass beakers 2 per sample plus 1 extra.
- Dedicated electrically operated end over end shaker (developed by ADAS).
- Temperature controlled environment at 20°C ± 2°C.
- Vacuum desiccator containing active silica gel.
- Stopwatch.
- Thermometer.
- Calgon dispersant (50g sodium hexametaphosphate and 7g anhydrous sodium carbonate in dissolved in distilled water and made up to 1 litre).
- Proforma sheet for data recording (example attached).

Analysis Procedure

The method used to determine aggregate stability is described by Anon (1982). This method compares the proportion, by weight, of silt and clay suspended by mild slaking forces to the total amount present in the sample. The test should be carried out in a temperature-controlled environment, since the rate at which particles settle out of suspension depends upon the temperature of the fluid.

Labelling

All samples will be labelled using the following method:

EQual YAS2002/SITE/PLOT NUMBER/DATE

Roles and Responsibilities

Analysis is to be conducted by a team led by the Laboratory Manager at ADAS Boxworth.

Data Recording

An entry will be made in the experimental diary to record the date of analysis and Analyst. The proforma in Appendix 1 will be used to record the results. Results will be manually transferred to an excel workbook and independently checked.

Health and Safety

- There is a risk of contracting tetanus from soil. Anyone who takes soil samples or working with soil should ensure they maintain a course of injections to protect themselves against tetanus infection. Protective clothing (goggles, gloves, laboratory clothing) should be worn and hands washed before eating, drinking or smoking.
- Instruments will only be operated by a trained member of the laboratory staff.
- A laboratory risk assessment for each analytical procedure is currently in place (which already considers aggregate stability) and will be consulted before and after analysis. If any amendments are required, the risk assessment will be updated.

References

Anon. (1982). *Techniques for Measuring Soil Physical Properties*. MAFF Reference Book 441. HMSO, London.

RECORD OF THE DETERMINATION OF SOIL STABILITY BY THE DISPERSION RATIO TEST

0.01 g Balance serial		0.0001 g Balance serial		Weight of beaker A	Weight of beaker A + calgon residue	Weight of calgon residue (a)
No.....checked*		No..... checked*				
YES	NO	YES	NO			

Aggregate size used	1 - 2 mm*	< 2 mm*	Other please specify
---------------------	-----------	---------	----------------------

Shaker calibration checked*		No. of inversions/minute set.
YES	NO	

* tick box

Lab No	Replicate No	Gentle Dispersion		
		Weight of beaker	Weight of beaker + residue	Weight of residue (b)
	1			
	2			
	3			
	1			
	2			
	3			
	1			
	2			
	3			
Recorded by and date				

Complete Dispersion			Dispersion Ratio
Weight of beaker	Weight of beaker + residue	Weight of residue (c)	

Certified by..... Location.....

Equal Project (30636) Method Statement:

Treatment Application: Poultry Litter Ash (PLA), Triple Superphosphate (TSP) & Muriate of Potash (MOP)

This method statement details the procedures to be followed when applying the Poultry Litter Ash (PLA), Triple Superphosphate (TSP) and Muriate of Potash (MOP) treatments at the two EQUAL field trials sites (ADAS Gleadthorpe and Harper Adams University) in years 1 (February 2013) and 2 (September/October 2013).

All treatments will be applied by hand at the same time at each experimental site (as far as practically possible). During application, samples of the PLA, TSP and MOP will be collected for laboratory analysis and archiving.

Equipment

- PLA, TSP and MOP pre-weighed into labelled bags for each plot
- 10 litre buckets
- String and pegs
- 1 litre plastic bottles
- 1 litre glass jars
- Boxes or crates for storage of the samples
- Experiment site plan.

Application Procedures

PLA, TSP and MOP will be applied in accordance with good agricultural practice (Defra, 2009; Defra, 2010) i.e. not when the soil is waterlogged or frozen, or if heavy rain is expected within 24 hours following application. The use of PLA must adhere to the PLA Quality Protocol (WRAP/EA, 2012).

- PLA will be applied at a rate of 700kg/ha; TSP at 187 kg/ha (equivalent to 84 kg/ha P₂O₅) and MOP at 140 kg/ha (equivalent to 84 kg/ha K₂O). Weigh the appropriate amount of PLA, TSP and MOP required for each plot and place in plastic bags.
- Locate the pre-weighed bags of product at the ends of each plot, carefully matching treatments and bags.
- Using string and pegs to mark out the plot boundaries.
- On each plot, empty the bag of product into a bucket and, holding the bucket, apply the product evenly to the relevant plots. This can be done by picking up a small handful of product and oscillating the hands horizontally whilst walking along and across the relevant plot at a uniform rate to give an even and complete coverage of the plot.
- Take representative samples of the PLA, TSP and MOP for laboratory analysis during spreading (*see section below*).
- Once applied, remove treatment markers.

Incorporation Procedures

In year 1, PLA, TSP and MOP will be top-dressed onto the growing winter wheat crop at Harper Adams, so will not be soil incorporated. At Gleadthorpe, the treatments will be applied to the stubble of the previous crop and will be incorporated prior to crop establishment. In year 2, the treatments at both sites will be applied to the stubble of the previous crop and soil incorporated prior to crop establishment. The method of incorporation (plough, tine or discs) will be in accordance with best practice for the site/soil type and will depend on ground conditions at the time of application and subsequent cropping. If ploughed, the depth of incorporation is typically c. 20cm, whereas discs tend to incorporate to a depth of c.15cm.

Sampling Procedures

Each year during PLA, TSP and MOP application at both field trials sites, representative samples will be taken and analysed at NLS as well as archived at Gleadthorpe, in accordance with the EA project specification '30636 EQual programme agriculture field trials Jan 2013':

- On the day of application, weigh out three replicate 2 kg samples of the PLA. Samples taken at Harper Adams should be sent to Gleadthorpe for further processing.
- At Gleadthorpe, split the sample in half and send 1 kg in a plastic tub to NLS for analysis of the adapted BioCompost analysis suite; store the remaining 1kg in glass jars for archiving in the cold store (<4°C).
- At Gleadthorpe only, on the day of application, weigh out a single c.0.5 kg sample of TSP and c.0.5 kg sample of MOP. Split each sample in half and send c.0.25 kg in an amber glass jar to NLS for analysis of the Dioxins suite; store the remaining c.0.25 kg in glass jars for archiving in the cold store (<4°C).
- Samples will be sent to NLS using overnight couriers (within 7 days of sampling). Archived samples will remain in the cold store at Gleadthorpe.

Labelling

Samples will be labelled for submission to NLS as detailed in the table below:

Label	Description
HAPLAPLA-1	PLA applied to block 1 at Harper Adams
HAPLAPLA-2	PLA applied to block 2 at Harper Adams
HAPLAPLA-3	PLA applied to block 3 at Harper Adams
GTPLAPLA-1	PLA applied to block 1 at Gleadthorpe
GTPLAPLA-2	PLA applied to block 2 at Gleadthorpe
GTPLAPLA-3	PLA applied to block 3 at Gleadthorpe
GTBAGTSP-	TSP 'out of the bag' applied at Gleadthorpe
GTBAGMOP-	MOP 'out of the bag' applied at Gleadthorpe

Additionally, the label should include the date of sampling.

Samples will be stored in a fridge (<4°C) prior to transit.

Roles and Responsibilities

Application and sampling of PLA, TSP and MOP is to be conducted by a team led by the Site Manager.

Data Recording

An entry will be made in the field trials diary to record the date and time of application and sampling, plots that treatments were applied to, application rates, weather and ground conditions at the time of application, sampler name and laboratory dispatch date and details.

Health and Safety

- It is advisable to wear protective/disposable gloves to minimise the risk of irritation to the skin and especially, to prevent fertiliser contacting open wounds. Wear an appropriate respirator to prevent inhalation of fine particles when handling in a confined space.
- Operators must comply with all health and safety instructions supplied with the product by manufacturers or suppliers.
- Leptospirosis (Weil's disease), a serious and sometimes fatal infection is transmitted to humans by contact with urine from infected rats. As rats often scavenge storage areas, take precautions as specified in HSE Agriculture Information Sheet no. 2.

- Wear a dust mask or preferably an airstream helmet when sampling dusty materials. Wash hands after using fertiliser before eating, drinking or smoking.
- Ensure that the outside of sample bags and containers are clean and free from contamination so that reception staff at the laboratory are not exposed to biohazards.
- The site specific risk assessment at each field trials location (which already considers fertiliser application and sampling) will be consulted before and after application. If any amendments are required, the risk assessment will be updated.

References

Defra (2009). *Protecting our Water, Soil and Air: A Code of Good Agricultural Practice for Farmers, Growers and Land Managers*. The Stationery Office, Norwich.

Defra (2010). *The Fertiliser Manual (RB209)*. The Stationery Office, Norwich

WRAP/EA (2012). *Quality Protocol - Poultry Litter Ash; End of waste criteria for the production and use of treated ash from the incineration of poultry litter, feathers and straw*.

EQual Project (30636) Method Statement:

Annual Soil Sampling & Analysis

This method statement details the procedures to be followed as part of the annual soil sampling programme at the two EQual field trials sites (ADAS Gleadthorpe and Harper Adams Agricultural College) during the study.

In Spring (March/April) 2013 & 2014 topsoil samples (0-15cm) will be taken from each plot at both field trials sites prior to the main manufactured fertiliser N applications and analysed to identify any treatment effects on selected soil and pore water properties.

Equipment

- Soil sampling for the chemical analyses will be taken using a hand-held "Cheese" corer. This is suitable for most soil conditions.
- A spade or trowel for aggregate stability sampling.
- Soil spatula, for use with cheese corer.
- Ruler graduated in cm.
- New clean polythene bags 300 x 200 mm to 500 x 400 mm, labelled accordingly.
- Rubber bands or ties (or use self-seal bags).
- Boxes for storage of samples.
- 1kg plastic tubs (supplied by NLS).
- 1 litre glass jars.
- Field trials site plan.

Sampling Procedures

The following samples should be taken when the soil is moist, but not frozen or too wet:

- Soil samples from 0-15cm depth will be taken following the methodology detailed in the "Fertiliser Manual (RB209)" (Defra, 2010; Appendix 3). Twenty soil core samples will be taken at even intervals in a 'W' shape pattern across each plot, using a hand held "Cheese corer", giving the specified volume of soil. The soil from each plot will be collected in one bag to generate one composite (representative) sample from each plot. Samples should be placed in 1 kg plastic tubs (3 per plot) and sent fresh in cool boxes to the NLS laboratory (store in a fridge at <4°C prior to dispatch).
- A further c.1kg of topsoil (0-15cm) will be collected according to the methodology detailed in the "Fertiliser Manual (RB209)" (Defra, 2010) for the determination of soil microbial biomass C & N. Twenty soil core samples will be taken at even intervals in a 'W' shape pattern across each plot, using a hand held "Cheese corer". The soil from each plot will be collected in one bag to generate one composite (representative) sample from each plot. Samples should be placed in plastic bags and sent fresh in cool boxes to the ADAS laboratory at Boxworth (F.A.O. Helen Kingston/Masuma Chauhan). Store in a fridge at <4°C prior to dispatch.
- Using a trowel/spade, twenty topsoil (0-15cm) samples will be taken from each plot spread evenly in a 'W' shape pattern to provide c.1.5kg of bulked fresh soil sample per plot. The soil sample should be sent to the ADAS laboratory at Boxworth (F.A.O. Helen Kingston/Masuma Chauhan) for the determination of aggregate stability. Care should be taken when sampling and during transit of these samples, in order to minimise disruption of the soil aggregates. Samples should be sent to the laboratory in boxes, and should not be stacked on top of each other.

- A further c.1kg topsoil (0-15cm) will be taken following the methodology detailed in the “Fertiliser Manual (RB209)” (Defra, 2010) for storage in the sample archive at ADAS Gleadthorpe. Samples should be stored in 1 litre glass jars.

Labelling

All samples will be labelled using the following method:

EQual YAS2002/SITE/PLOT NUMBER/DATE

Samples will not be dispatched on a Friday, as they may deteriorate over the weekend. Samples will be stored in a fridge (<4°C) prior to transit.

Roles and Responsibilities

Sampling is to be conducted by a team led by the Site Manager

Data Recording

An entry will be made in the field trials diary to record the date of soil sampling, weather and ground conditions, plots that were sampled, sampler name and laboratory dispatch date and details.

Health and Safety

- There is a risk of contracting tetanus from soil. Anyone who takes soil samples should ensure they maintain a course of injections to protect themselves against tetanus infection.
- Efficient soil sampling tools have sharp edges; care should be taken when they are used.
- Back injuries can occur when using soil sampling tools. This risk can be minimised by using a good lifting technique, i.e. keep the back straight and bend the knees.
- The site specific risk assessment at each field trials location (which already considers soil sampling) will be consulted before and after sampling. If any amendments are required, the risk assessment will be updated.

References

Defra (2010). The Fertiliser Manual (RB209) 8th edition. The Stationery Office, Norwich

EQual Project (30636) Method Statement:

Harvest – Grain/seed yield & analysis

This method statement details the procedures to be followed for harvesting the two EQual field trials sites (ADAS Gleadthorpe and Harper Adams Agricultural College) in August 2013 & 2014 for the determination of crop yield, dry matter content, nutrient offtake and uptake of heavy metals and organic contaminants.

Materials and Equipment

- Experiment site plan.
- Record sheet and pen.
- Polypropylene screw top bottles of 1 litre capacity, labelled accordingly
- A plastic stacking tray or other suitable container to store the bottles whilst harvesting is in progress.
- Dust mask and safety glasses or Airstream helmet (for operators not protected by a cab).
- Ear plugs

Harvesting and Sampling Procedure

Harvesting of plots will take place in line with good agricultural practice (Defra, 2009; Defra, 2010) i.e. when the soil is dry enough to support the weight of a combine thus reducing the risks of damaging the soil structure and causing compaction. A small plot combine will be used, driven by an experienced member of staff. Record the weight of grain harvested from each plot and the harvest area (plot length x harvest width).

During harvesting, c.1 kg samples of the grain from each individual plot will be collected in a 1 litre polypropylene screw top bottle labelled accordingly. Take the sample by gradually filling the bottle from the flowing grain sample 'little and often'.

Prior to dispatching all grain/seed samples to the laboratory for analysis, a c. 100g sub-sample should be taken from each plot for immediate dry matter determination (by drying a known weight of sample for 24 hours at 100°C; the dry matter content is then calculated from the difference in weight between the fresh and dry grain/seed sample). The remaining grain will be sent to NLS to analyse the grain nutrient, metal and organic contaminant concentrations.

Labelling

All samples will be labelled using the following method:

EQual YAS2002/SITE/PLOT NUMBER/DATE

Roles and Responsibilities

Harvesting is to be conducted by a team led by the Site Manager

Data Recording

An entry will be made in the field trials diary to record the date of harvesting, weather and field conditions, sampler name and laboratory dispatch date and details.

The weight of grain harvested from each plot, together with the harvest area (plot length x harvested width) should be recorded on a standard proforma (Appendix 1).

Health and Safety

- Most combines in use in ADAS have cabs for the driver, but in some cases the operator(s) is/are exposed. In the absence of cab protection, operators should wear an approved dust mask with protective glasses or an air-stream type helmet. The Health & Safety Executive Agricultural Sheet No 3 gives the maximum exposure limit for grain dust as specified in the Schedule of COSHH Regulations 1988.
- Combine harvesters are noisy when operating; wear either approved ear plugs or ear muffs where noise is above recommended exposure levels.
- A site specific risk assessment for each field trials location is currently in place (which already considers harvesting) and will be consulted before and after analysis. If any amendments are required, the risk assessment will be updated.

References

Defra (2009) *Protecting our Water, Soil and Air: A Code of Good Agricultural Practice for Farmers, Growers and Land Managers*. The Stationary Office, Norwich.

Defra (2010). *The Fertiliser Manual (RB209) 8th edition*. The Stationery Office, Norwich

Standard record proforma

ADAS GLEADTHORPE

Scale weight checked

Trial no. Year :-..... G.S.:-..... Sheet of

Site:- S.O.P. no :- Date :-

Obs./Assessment :-

Area/Unit/Scale :-

M=mistake in recording
 C=calculation error
 P=recorded in wrong place
 T=transcription error
 CH= e.g. checked(unusual result has been checked)
 E=error explained elsewhere

Plot																		
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
0																		
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
0																		

Balance I.D. no.
 Drying oven I.D. no.
 Recorded by:-
 Date:-

Certified by:-
 Date:-

d:\data\crops\2011\record.xls

Appendix 2. Field trials site diaries and fertiliser inputs

Gleadthorpe field trials diary and fertiliser policy

Activity	Date
Site characterisation soil sample	28/1/13
5 t/ha lime and 100 kg/ha K ₂ O applied to all treatments	7/2/13
Application of PLA, TSP and MOP treatments	13/2/13
Spring barley drilled	8/4/13
40 kg/ha N and 50 kg/ha SO ₃ applied to all treatments	15/4/13
70 kg/ha N and 3 kg/ha MnSO ₄ applied to all treatments	4/5/13
3 kg/ha MnSO ₄ applied to all treatments	1/6/13
3 kg/ha MnSO ₄ applied to all treatments	25/6/13
Spring barley harvested	5/9/13
Application and incorporation of PLA, TSP and MOP treatments	12/11/13
Winter wheat drilled	2/12/13
100 kg/ha K ₂ O and 40 kg/ha N applied to all treatments	20/2/14
Annual soil sample	28/4/14
60 kg/ha N, 50 kg/ha SO ₃ and 4.5 kg/ha MnSO ₄ to all treatments	29/4/14
60 kg/ha N and 2.5 kg/ha MnSO ₄ to all treatments	15/5/14
Winter wheat harvested	13/8/14

Harper Adams field trials diary and fertiliser policy

Activity	Date
Site characterisation soil sample	30/1/13
Application of PLA, TSP and MOP treatments	26/2/13
40 kg/ha N, 75 kg/ha K ₂ O and 40 kg/ha SO ₃ applied to all treatments	3/4/13
70 kg/ha N applied to all treatments	26/4/13
70 kg/ha N applied to all treatments	16/5/13
Winter wheat harvested	21/8/13
Application and incorporation of PLA, TSP and MOP treatments	7/10/13
Spring barley drilled	13/3/13
110 kg/ha N applied to all treatments	8/4/14
Annual soil sample	28/4/14
35kg/ha K ₂ O, 50 kg/ha SO ₃ & 5 kg/ha MnSO ₄ to all treatments	6/5/14
Winter wheat harvested	7/8/14

Appendix 3: Storage Study Method Statement

EQual Project (30636) Method Statement:

Storage study

This method statement details the procedures to be followed for the storage study being undertaken at ADAS Gleadthorpe.

In order to determine the effects of storage on the chemical composition of poultry litter ash (PLA) and paper sludge (PS) materials replicated storage heaps will be established at ADAS Gleadthorpe in August 2013 and changes in the composition of the stored materials will be monitored over the course of 12 months. Heaps will be established on a slightly sloping, impermeable base in order to collect and analyse any leachate generated from the heaps.

The storage and use of PS must adhere to the requirements of the EA Regulatory Position Statement. The storage and use of PLA must adhere to the PLA Quality Protocol.

Equipment

- Polythene liners, straw bales, drainpipes and tanks to enable leachate collection
- Portable weigh pads
- Tinytalk temperature data loggers
- Litter bags, ties, string and waterproof labels
- 1kg plastic tubs (supplied by NLS)
- 120g glass jars (supplied by NLS)
- 300g polypropylene sterile jars (supplied by NLS)
- 1 litre glass jars
- Bottles for leachate (supplied by NLS)
- Camera

Heap construction

Six individual temporary storage heaps (3 replicated PLA heaps & 3 replicated PS heaps) will be established in a series of hydrologically isolated, sloping concrete bunkers at ADAS Gleadthorpe. Each bunker will be 3.625m x 3.625m in plan, with two courses of concrete blocks around three of the sides (c.0.5m high), and the fourth side contained using straw bales lined with a strip of polythene. Leachate will be collected at the lowest corner of each bunker using a short length of perforated plastic drainage pipe to direct leachate into six individual collection tanks.

Approximately 5 tonnes (fresh weight - FW) of PLA and PS material will be weighed into the bays using portable weigh pads. During construction four 'litter' bags, each containing c.2kg of the PLA or PS material will be buried at known, separate locations within the heap. Each litter bag will have a string attached to aid retrieval, with the location of each bag identified by markers on the surface of the heap. Additionally, a tinytalk temperature data logger will be placed in each heap, set to record the heap temperature on an hourly basis.

Photographs will be taken on heap construction and at each sampling occasion. The heaps will be dismantled at the end of the 12 month storage period.

Sampling

Representative samples will be taken for laboratory analysis at NLS and archiving at Gleadthorpe, in accordance with the EA project specification '30636 EQal programme agriculture field trials Jan 2013':

Samples of the material contained within each heap will be taken on construction in August 2013 (3 samples of each material, c.2kg per sample). Samples will be split and placed into the appropriate sample bottles, and sent to NLS for analysis.

The litter bags will be removed at 1, 3, 6 and 12 months after heap construction. Samples within the litter bags will be split and placed in the appropriate sample bottles and sent to NLS for analysis.

Samples for e-coli and salmonella analysis should be dispatched to NLS on the same day of sampling. A subsample of each material will also be placed in glass jars for storage in the archive at Gleadthorpe.

Leachate from each heap will be collected in a series of prefabricated, calibrated containers of appropriate size. If leachate arises, the volume collected will be measured at fortnightly/monthly intervals (up to a maximum of 12 sampling dates and therefore 6x12=72 samples over the period September 2013 to August 2014, depending on rainfall). Samples will be taken from the collected bulk of leachate from each heap, will be split and placed into the appropriate sample bottles, and sent to NLS for analysis.

All samples should be stored in a fridge (<4°C) and sent, fresh, in a cool box (with ice packs) via overnight carrier or equivalent. Do not dispatch samples on a Friday, as they may deteriorate over the weekend. Please inform NLS of their dispatch and expected time of arrival. All glass bottles for sample storage should be washed (i.e. in a normal dishwasher using clean water only) before use, to remove any residues.

Labelling

Samples will be labelled with a unique identifier as detailed in Table 1.

Table 1 Sample labelling

Treatment (heap)	Sample type	Reference label ^a
PLA rep 1	PLA material	SSPLAPLA-1 <i>Sample date</i>
PLA rep 2	PLA material	SSPLAPLA-2 <i>Sample date</i>
PLA rep 3	PLA material	SSPLAPLA-3 <i>Sample date</i>
Paper sludge rep 1	PS material	SSPSFPSF-1 <i>Sample date</i>
Paper sludge rep 2	PS material	SSPSFPSF-2 <i>Sample date</i>
Paper sludge rep 3	PS material	SSPSFPSF-3 <i>Sample date</i>
PLA rep 1	Leachate	SSPLALEAC1 <i>Sample date</i>
PLA rep 2	Leachate	SSPLALEAC2 <i>Sample date</i>
PLA rep 3	Leachate	SSPLALEAC3 <i>Sample date</i>
Paper sludge rep 1	Leachate	SSPSFLEAC1 <i>Sample date</i>
Paper sludge rep 2	Leachate	SSPSFLEAC2 <i>Sample date</i>
Paper sludge rep 3	Leachate	SSPSFLEAC3 <i>Sample date</i>

^aThe reference label should be followed by the date of sampling

Roles and Responsibilities

All activities will be conducted by a team led by the Site Manager

Data Recording

An entry will be made in the field trials diary to record the date and time of heap construction, and all sampling occasions, the weather conditions, sampler name and laboratory dispatch date and details. Heap temperatures at a single point in the 'centre' of each heap will be recorded on a daily basis using temperature logging equipment. Weather variables routinely monitored by the onsite meteorological station at Gleadthorpe will be used to assist in interpretation of results, in particular daily rainfall and ambient air temperatures.

Health and Safety

- Avoid getting the material on the skin, but if this happens, wash it off as soon as possible. Wash hands and forearms thoroughly before handling food, drinking or smoking.
- Leptospirosis (Weil's disease), a serious and sometimes fatal infection is transmitted to humans by contact with urine from infected rats. As rats often scavenge organic material stores, take precautions as specified in HSE Agriculture Information Sheet no. 2.
- Handling organic material may expose workers to other serious infectious diseases. These include poliomyelitis, tetanus, typhoid fever, hepatitis A and hepatitis B. See Occupational Health Service Notice 93/6.
- Wear waterproof boots, waterproof jacket, trousers and gauntlets or long gloves, when sampling organic materials. Wear a face shield where splashing of material may occur. Wear a dust mask or preferably an airstream helmet when sampling dusty materials.
- Ensure that the outside of sample bags and containers are clean and free from contamination so that reception staff at the laboratory are not exposed to biohazards.
- The site specific risk assessment at each field trials location (which already considers organic material application and sampling) will be consulted before and after application. If any amendments are required, the risk assessment will be updated.

Appendix 4: Topsoil properties April 2013 & 2014

Topsoil chemical properties following the first annual addition of PLA and P&K, -April 2013
(treatment means)

Determinand	Unit	Gleadthorpe				Harper Adams			
		Con	PLA	P&K	P ¹	Con	PLA	P&K	P ¹
Conductivity	µs/cm	2033 ^b	1977 ^a	1990 ^a	0.08	2033	2087	2037	NS (0.35)
pH	unit	6.50	6.54	6.45	NS (0.95)	6.77	7.57	7.13	NS (0.26)
Extractable P	mg/kg dm	48.3	49.6	45.7	NS (0.82)	78.4	89.0	89 ²	NS (0.73)
Extractable K	mg/kg dm	90.7 ^a	86.1 ^a	101.7 ^b	0.003	135	187	183	NS (0.10)
Extractable Mg	mg/kg dm	65.9 ^b	47.6 ^a	42.3 ^a	0.026	48.7	54.4	50.3	NS (0.69)
Aluminium	mg/kg dm	5223	5347	5280	NS (0.94)	11500	11633	10800	NS (0.42)
Antimony	mg/kg dm	< 1.0	1.11	1.05	NS (0.41)	1.07	1.17	1.04	NS (0.74)
Arsenic	mg/kg dm	4.58	4.63	4.70	NS (0.85)	6.20	6.36	6.10	NS (0.72)
Barium	mg/kg dm	25.9	24.0	25.2	NS (0.46)	63.7	66.8	61.1	NS (0.57)
Beryllium	mg/kg dm	0.35	0.37	0.37	NS (0.33)	0.51	0.55	0.54	NS (0.84)
Boron	mg/kg dm	3.92	4.38	3.90	NS (0.68)	8.21	7.88	7.59	NS (0.57)
Cadmium	mg/kg dm	< 0.2	< 0.2	< 0.2	NS (1.0)	<0.2	<0.2	<0.2	NS (1.0)
Calcium	mg/kg dm	1008	973	889	NS (0.83)	2240	3840	3153	NS (0.30)
Chromium	mg/kg dm	7.23	6.93	7.03	NS (0.66)	14.2	14.9	13.1	NS (0.47)
Chromium VI	mg/kg dm	< 0.3	< 0.3	< 0.3	NS (1.0)	<0.3	<0.3	<0.3	NS (1.0)
Cobalt	mg/kg dm	1.83	1.78	1.80	NS (0.83)	3.86	4.21	3.65	NS (0.35)
Copper	mg/kg dm	5.18	5.12	5.14	NS (0.99)	12.8	14.6	12.8	NS (0.12)
Iron	mg/kg dm	8360	8180	8387	NS (0.87)	11600	11760	11067	NS (0.78)
Lead	mg/kg dm	16.0 ^c	14.8 ^a	15.5 ^b	<0.001	17.6	18.4	17.2	NS (0.23)
Lithium	mg/kg dm	6.62	7.19	7.00	NS (0.59)	15.0	17.3	14.7	NS (0.14)
Magnesium	mg/kg dm	742	796	735	NS (0.64)	2533	2703	2397	NS (0.24)
Manganese	mg/kg dm	202 ^b	141 ^a	158 ^a	<0.001	285	291	263	NS (0.59)
Mercury	mg/kg dm	< 0.2	< 0.2	< 0.2	NS (1.0)	<0.2	<0.2	<0.2	NS (1.0)
Molybdenum	mg/kg dm	< 1.0	< 1.0	< 1.0	NS (1.0)	<1.0	<1.0	<1.0	NS (1.0)
Nickel	mg/kg dm	4.15	4.52	4.41	NS (0.51)	10.1	11.7	9.59	NS (0.31)
Phosphorus	mg/kg dm	639	592	598	NS (0.57)	1060	1280	1110	NS (0.09)
Potassium	mg/kg dm	744	779	760	NS (0.67)	2287	2520	2227	NS (0.23)
Selenium	mg/kg dm	< 1.0	< 1.0	< 1.0	NS (1.0)	<1.0	<1.0	<1.0	NS (1.0)
Silver	mg/kg dm	< 1.0	< 1.0	< 1.0	NS (1.0)	<1.0	<1.0	<1.0	NS (1.0)
Sodium	mg/kg dm	31.8 ^a	41.6 ^b	38.2 ^{ab}	0.03	71.3 ^a	113 ^b	68.5 ^a	0.04
Strontium	mg/kg dm	4.80	5.22	5.04	NS (0.67)	8.66 ^a	12.3 ^b	10.2 ^a	0.02
Thallium	mg/kg dm	< 1.0	< 1.0	< 1.0	NS (1.0)	<1.0	<1.0	<1.0	NS (1.0)
Tin	mg/kg dm	1.08	1.06	1.09	NS (0.43)	1.33	1.39	1.31	NS (0.86)

Titanium	mg/kg dm	79.6	77.4	78.2	NS (0.88)	109	115	99.1	NS (0.20)
Vanadium	mg/kg dm	11.1	10.8	11.0	NS (0.79)	17.9	18.5	16.8	NS (0.53)
Zinc	mg/kg dm	28.5	29.4	26.4	NS (0.54)	61.4	71.9	61.0	NS (0.25)
Biomass C ³	mg/kg	37	34	29	NS (0.54)	178	199	181	NS (0.33)
Biomass N	mg/kg	15	19	11	NS (0.18)	28	30	29	NS (0.89)
Aggregate stability ⁴	%w/w	15	16	16	NS (0.62)	10	7	11	NS (0.21)

¹Statistical analysis undertaken using ANOVA (data normally distributed); Numbers in brackets indicate the P statistic; ^{a,b}Different letters between columns indicate significant differences between treatments for a particular site at $P < 0.05$; NS=not significant ($P > 0.05$).²Mean of two replicate plots; ³Note a number of analysis values were at or close to the limits of analytical detection; ⁴Aggregate stability measured by the dispersion ratio (ratio of silt and clay suspended by mild slaking forces expressed as a % of the total silt and clay content); Ratios in the range 6-10% suggest the soil is 'stable', 11-15% indicate that the soil is 'fairly stable', while ratios in the range 16-25% suggest the soil is 'somewhat unstable' (Anon, 1982).

**Topsoil chemical properties following two years of PLA and P&K additions, April 2014
(treatment means)**

Determinand	Unit	Gleadthorpe				Harper Adams			
		Con	PLA	P&K	P ¹	Con	PLA	P&K	P ¹
Conductivity	µs/cm	2120	2113	2143	NS (0.44)	2123	2150	2133	NS (0.07)
pH	unit	6.20	6.64	6.75	NS (0.17)	6.45	6.95	6.62	NS (0.50)
Extractable P	mg/kg dm	43.3 ^a	59.9 ^d	48.6 ^a	0.013	89.6	82.7	89.1	NS (0.68)
Extractable K	mg/kg dm	62.8	74.3	88.2	NS (0.09)	119	177	156	NS (0.39)
Extractable Mg	mg/kg dm	62.3 ^d	44.0 ^a	40.0 ^a	0.018	48.0	51.2	50.3	NS (0.92)
Aluminium	mg/kg dm	4453	4813	4483	NS (0.26)	11367	10500	11400	NS (0.07)
Antimony	mg/kg dm	<1	<1	<1	NS (1.0)	1.03	<1	<1	NS (1.0)
Arsenic	mg/kg dm	4.52	4.32	4.45	NS (0.83)	6.89	6.16	6.45	NS (0.37)
Barium	mg/kg dm	22.1	22.3	20.7	NS (0.08)	64.2 ^b	57.5 ^a	61.0 ^a	0.012
Beryllium	mg/kg dm	0.31	0.36	0.33	NS (0.08)	0.56	0.52	0.55	NS (0.42)
Boron	mg/kg dm	2.64	2.92	2.59	NS (0.64)	8.70	7.11	8.31	NS (0.18)
Cadmium	mg/kg dm	<0.2	<0.2	<0.2	NS (1.0)	0.24	0.23	0.23	NS (0.14)
Calcium	mg/kg dm	1006	1208	1441	NS (0.47)	2610	2400	2757	NS (0.66)
Chromium	mg/kg dm	6.70	7.02	6.35	NS (0.19)	15.4	14.8	15.1	NS (0.65)
Chromium VI	mg/kg dm	<0.6	<1.2	<1.2	NS (1.0)	<0.6	<0.6	<0.6	NS (1.0)
Cobalt	mg/kg dm	1.84	1.94	1.74	NS (0.13)	4.57	4.02	4.26	NS (0.20)
Copper	mg/kg dm	4.27 ^a	4.92 ^b	4.40 ^a	0.03	13.2	12.9	13.6	NS (0.11)
Iron	mg/kg dm	7040	7373	6927	NS (0.38)	12133	10767	11467	NS (0.15)
Lead	mg/kg dm	15.8	14.9	14.3	NS (0.29)	19.9	18.7	18.5	NS (0.17)
Lithium	mg/kg dm	5.91	6.79	6.10	NS (0.07)	15.2	13.8	14.9	NS (0.09)
Magnesium	mg/kg dm	671	724	633	NS (0.52)	2443	2280	2443	NS (0.21)
Manganese	mg/kg dm	185 ^b	136 ^a	130 ^a	0.02	312 ^b	269 ^a	276 ^a	0.05
Mercury	mg/kg dm	<0.2	<0.2	<0.2	NS (1.0)	<0.2	<0.2	<0.2	NS (1.0)
Molybdenum	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Nickel	mg/kg dm	3.97 ^a	4.60 ^b	4.20 ^{ab}	0.05	11.4	11.2	11.3	NS (0.88)
Phosphorus	mg/kg dm	514	566	549	NS (0.35)	1167	1100	1217	NS (0.52)
Potassium	mg/kg dm	656	697	654	NS (0.59)	2263 ^b	2090 ^a	2357 ^b	0.015
Selenium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Silver	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Sodium	mg/kg dm	23.7	24.8	21.7	NS (0.30)	62.6	60.7	69.4	NS (0.38)
Strontium	mg/kg dm	4.25	5.02	5.52	NS (0.28)	9.80	9.24	9.42	NS (0.62)
Thallium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Tin	mg/kg dm	<1	1.0	1.0	NS (1.0)	1.32	1.23	1.57	NS (0.12)
Titanium	mg/kg dm	77.6	74.8	73.8	NS (0.47)	119	115	115	NS (0.12)
Vanadium	mg/kg dm	10.0	10.4	9.7	NS (0.34)	19.2	17.8	19.0	NS (0.08)

Zinc	mg/kg dm	25.0	26.1	23.8	<i>NS (0.24)</i>	65.3	66.5	65.0	<i>NS (0.83)</i>
Sum 7 PCBs	mg/kg dm	0.0002	0.0002	0.0003	<i>NS (0.64)</i>	nd	nd	nd	<i>nd</i>
Sum 12 PCBs TEQ	ng/kg dm	0.0004	0.0004	0.0004	<i>NS (0.52)</i>	nd	nd	nd	<i>nd</i>
Dioxins and furans TEQ	ng/kg dm	3.19	4.28	4.33	<i>NS (0.27)</i>	nd	nd	nd	<i>nd</i>
Biomass C	mg/kg	55	32	49	<i>NS (0.07)</i>	179	238	144	<i>NS (0.06)</i>
Biomass N	mg/kg	18	13	13	<i>NS (0.18)</i>	26	29	28	<i>NS (0.34)</i>
Aggregate stability ³	%w/w	16	18	19	<i>NS (0.16)</i>	7	9	10	<i>NS (0.41)</i>

¹Statistical analysis undertaken using ANOVA (data normally distributed); Numbers in brackets indicate the P statistic; ^{a,b}Different letters between columns indicate significant differences between treatments for a particular site at $P < 0.05$; *NS*=not significant ($P > 0.05$); ³Aggregate stability measured by the dispersion ratio (ratio of silt and clay suspended by mild slaking forces expressed as a % of the total silt and clay content); Ratios in the range 6-10% suggest the soil is 'stable', 11-15% indicate that the soil is 'fairly stable', while ratios in the range 16-25% suggest the soil is 'somewhat unstable' (Anon, 1982).

Changes in soil properties over time Jan 13 – April 13 – April 14: ANOVA P statistics (significance of time, treatment & the interaction between treatment & time; including the direction of change)¹

<i>Determinand</i>	<i>Gleadthorpe</i>				<i>Harper Adams</i>			
	<i>Time</i>	<i>Treat</i>	<i>Trt x time</i>	\updownarrow^2	<i>Time</i>	<i>Treat</i>	<i>Trt x time</i>	\updownarrow^1
Conductivity	<0.001	0.001	0.016	↑	<0.001	NS	0.04	↑
pH	<0.001	NS	NS	↑	0.05	0.05	NS	∩
Extractable P	NS	NS	0.05	↑	NS	NS	NS	
Extractable K	<0.001	0.05	NS	∩	NS	NS	NS	
Extractable Mg	NS	<0.001	NS		NS	NS	NS	
Aluminium	NS	NS	NS		NS	NS	NS	
Arsenic	NS	NS	NS		NS	NS	NS	
Barium	NS	NS	NS		NS	NS	NS	
Beryllium	0.03	NS	NS	∩	NS	NS	NS	
Boron	<0.001	NS	NS	∩	NS	NS	NS	
Calcium	0.025	NS	NS	↑	NS	NS	NS	
Chromium	NS	NS	NS		NS	NS	NS	
Cobalt	NS	NS	NS		0.04	NS	NS	↑
Copper	0.008	NS	NS		NS	NS	NS	
Iron	0.002	NS	NS	∩	NS	NS	NS	
Lead	NS	NS	NS		0.007	NS	NS	↑
Lithium	NS	NS	NS		NS	NS	NS	
Magnesium	NS	NS	NS		NS	NS	NS	
Manganese	NS	<0.001	NS		NS	NS	NS	
Nickel	NS	NS	NS		0.01	NS	NS	
Phosphorus	NS	NS	NS		NS	NS	NS	
Potassium	0.02	NS	NS	∩	NS	NS	NS	
Sodium	<0.001	0.05	NS	∩	0.006	0.002	0.01	∩
Strontium	NS	NS	NS		NS	0.02	NS	
Titanium	<0.001	NS	NS	∩	<0.001	0.02	0.03	↑
Vanadium	NS	NS	NS		NS	NS	NS	
Zinc	NS	NS	NS		NS	0.02	0.53	
Biomass C	<0.001	0.03	NS	-	NS	NS	NS	
Biomass N	NS	NS	NS		0.01	NS	NS	↓
Aggregate stability	NS	0.01	NS		NS	NS	NS	

¹NS=not significant ($P>0.05$); ²∩General direction of change: ↑ Increase over time; ↓ Decrease over time; ∩ increase to April 2013 followed by a decrease to background levels

Appendix 5: Grain nutrient and metal concentrations: harvests 2013 & 2014

Grain nutrient and metal concentrations at harvest 2013

<i>Determinand</i>	<i>Unit</i>	<i>Gleadthorpe (Spring barley)</i>				<i>Harper Adams (Winter wheat)</i>			
		<i>Con</i>	<i>PLA</i>	<i>P&K</i>	<i>P¹</i>	<i>Con</i>	<i>PLA</i>	<i>P&K</i>	<i>P¹</i>
Aluminium	mg/kg dm	<50	<50	<50	NS (1.0)	<50	<50	<50	NS (1.0)
Antimony	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Arsenic	mg/kg dm	0.71	0.56	0.58	NS (0.31)	0.61	0.61	0.62	NS (0.86)
Barium	mg/kg dm	8.61	7.40	5.49	NS (0.67)	1.99	2.32	2.15	NS (0.88)
Beryllium	mg/kg dm	<0.1	<0.1	<0.1	NS (1.0)	<0.1	<0.1	<0.1	NS (1.0)
Boron	mg/kg dm	5.45	4.60	3.32	NS (0.50)	3.07	3.17	3.40	NS (0.93)
Cadmium	mg/kg dm	0.29	<0.2	<0.2	NS (1.0)	<0.2	<0.2	<0.2	NS (1.0)
Calcium	mg/kg dm	1123	854	666	NS (0.23)	596	565	575	NS (0.90)
Chromium	mg/kg dm	4.06	1.66	0.62	NS (0.46)	<0.5	<0.5	0.48	NS (1.0)
Cobalt	mg/kg dm	3.07	3.34	0.46	NS (0.55)	1.75	0.13	1.33	NS (0.35)
Copper	mg/kg dm	4.51	3.10	2.75	NS (0.35)	2.45	2.45	2.45	NS (1.0)
Iron	mg/kg dm	<200	<200	<200	NS (1.0)	<200	<200	<200	NS (1.0)
Lead	mg/kg dm	4.23	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Lithium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Magnesium	mg/kg dm	1065	972	1062	NS (0.87)	875	822	897	NS (0.41)
Manganese	mg/kg dm	31.5	17.5	15.7	NS (0.24)	24.4	21.9	25.8	NS (0.75)
Mercury	mg/kg dm	<0.2	<0.2	<0.2	NS (1.0)	<0.2	<0.2	<0.2	NS (1.0)
Molybdenum	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Nickel	mg/kg dm	2.44	1	0.3	NS (0.47)	<0.6	<0.6	<0.6	NS (1.0)
Phosphorus	mg/kg dm	2803	2993	3540	NS (0.77)	3463	3430	3843	NS (0.38)
Potassium	mg/kg dm	7390	6953	5923	NS (0.61)	4330	4543	4767	NS (0.45)
Selenium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Silver	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Sodium	mg/kg dm	88.8	87.2	42.2	NS (0.45)	11.4	13.9	13.3	NS (0.83)
Strontium	mg/kg dm	3.86	3.13	2.31	NS (0.33)	1.06	1.20	1.44	NS (0.95)
Thallium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Tin	mg/kg dm	1.67	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Titanium	mg/kg dm	5.00	2.33	1.5	NS (0.24)	<3	<3	<3	NS (1.0)
Vanadium	mg/kg dm	0.59	0.11	0.05	NS (0.22)	<0.1	<0.1	<0.1	NS (1.0)
Zinc	mg/kg dm	40.8	31.8	30.4	NS (0.3)	30.2	31.6	35.4	NS (0.77)

¹Statistical analysis undertaken using ANOVA (data normally distributed); NS=not significant ($P>0.05$)

Grain nutrient and metal concentrations at harvest 2014

Determinand	Unit	Gleadthorpe (winter wheat)				Harper Adams (spring barley)			
		Con	PLA	P&K	P ¹	Con	PLA	P&K	P ¹
Aluminium	mg/kg dm	<50	74.3	<50	NS (1.0)	59.5	<50	<50	NS (1.0)
Antimony	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Arsenic	mg/kg dm	1.08	1.10	1.12	NS (0.77)	1.14	1.12	1.08	NS (0.67)
Barium	mg/kg dm	4.41	5.70	5.59	NS (0.23)	2.32	2.07	1.44	NS (0.39)
Beryllium	mg/kg dm	<0.1	<0.1	0.15	NS (1.0)	<0.1	0.18	<0.1	NS (1.0)
Boron	mg/kg dm	2.77	2.70	2.03	NS (0.06)	3.09	2.33	2.24	NS (0.62)
Cadmium	mg/kg dm	<0.2	<0.2	<0.2	NS (1.0)	<0.2	<0.2	<0.2	NS (1.0)
Calcium	mg/kg dm	440	589	447	NS (0.36)	712	604	542	NS (0.27)
Chromium	mg/kg dm	<0.5	0.50	<0.5	NS (1.0)	<0.5	0.52	<0.5	NS (1.0)
Cobalt	mg/kg dm	<0.1	<0.1	<0.1	NS (1.0)	<0.1	<0.1	<0.1	NS (1.0)
Copper	mg/kg dm	2.37	2.30	1.97	NS (0.09)	2.17	2.25	1.7	NS (0.29)
Iron	mg/kg dm	<200	227	<200	NS (1.0)	<200	<200	<200	NS (1.0)
Lead	mg/kg dm	<1	2.80	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Lithium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Magnesium	mg/kg dm	995	995	986	NS (0.99)	876	830	782	NS (0.54)
Manganese	mg/kg dm	29.1	29.3	31.5	NS (0.83)	12.4	12.2	11.1	NS (0.79)
Mercury	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Molybdenum	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Nickel	mg/kg dm	<0.6	<0.6	<0.6	NS (1.0)	<0.6	<0.6	<0.6	NS (1.0)
Phosphorus	mg/kg dm	3433	3460	3480	NS (0.98)	3553	3757	3490	NS (0.64)
Potassium	mg/kg dm	4957	4980	4910	NS (0.60)	5533	5490	5000	NS (0.22)
Selenium	mg/kg dm	1.08 ^a	1.30 ^b	1.29 ^b	0.012	1.26	1.31	1.33	NS (0.57)
Silver	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Sodium	mg/kg dm	13.4	22.0	16.7	NS (0.24)	53.2	54.4	30.1	NS (0.13)
Strontium	mg/kg dm	1.89	2.6	2.30	NS (0.19)	1.52	1.24	1.09	NS (0.49)
Thallium	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Tin	mg/kg dm	<1	<1	<1	NS (1.0)	<1	<1	<1	NS (1.0)
Titanium	mg/kg dm	<3	3.50	<3	NS (1.0)	<3	<3	<3	NS (1.0)
Vanadium	mg/kg dm	<0.1	0.20	0.10	NS (0.22)	0.12	0.12	<0.1	NS (0.08)
Zinc	mg/kg dm	33.0	31.1	28.9	NS (0.37)	24.3	27.3	24.5	NS (0.32)

¹Statistical analysis undertaken using ANOVA (data normally distributed); ^{a,b}Different letters between columns indicate significant differences between treatments for a particular site at $P < 0.05$; NS=not significant ($P > 0.05$)