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PARAQUAT PERSISTENCE - STATISTICAL ANALYSIS OF THE W.R.O LONG TERM TRIAL

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1 INTRODUCTION

A trial has been in progress at WRO since 1967. This trial has been described in several publications (references 1-3). Over the whole period there have been basically four treatments with three replicate plots each. These are:

Treatment	Dose per Application kg/ha	No. of Applications per Year	Target
PQ1	1.12	4	Soil
PQ2	4.48	1	Soil
PQ3	1.12	4	Vegetation
PQ4*	4.48	1	Vegetation

*The trial was slightly more complicated originally as described in the references, this treatment was first applied only in 1969. It was untreated previously.

Soil samples were taken to a depth of 36 centimetres in 1971, 1973, 1975 and then annually up to and including 1983. The samples were taken in the spring of each of these years just prior to the first application of paraquat for that year. Thus the 1971 samples were preceded by 4 years applications (except for PQ4). These samples have been analysed for paraquat residues.

This report details the statistical analysis of this data with the objective of determining:

1) Whether there was evidence of paraquat degradation
2) Estimating the degradation rate if appropriate

2 DATA

The soil residue data obtained are given in Appendix 1. The individual plot data were not available for 1971 and for PQ1 and PQ3 the mean of the incorporated and non-incorporated (see reference 1) have been used. The treatment means have been used in the analysis against time reported later.

The applicates dates are given in Appendix 2. The first date in each year applied for PQ2 and PQ4 (except 1967 and 1968). In brackets under the dates in this appendix are the estimated number of years since the first application in 1967.

3 ANALYSIS

When paraquat is applied, particularly onto vegetation, it is not known how much paraquat reaches and becomes bound in the soil. So although we know the application rates we do not know the actual amounts added to the soil each time. Approximately, however, we would expect the proportion reaching the soil to be constant from year to year. We would certainly not expect this proportion to vary in a systematic way with time nor to be reduced by the level of residue in the soil (in this trial).

Therefore, if paraquat does not degrade, the paraquat soil residues would be expected to increase from zero in 1967 by a fixed annual increment. In other words, the data would follow a linear relation passing through zero in spring 1967. If paraquat degrades at all then the residues will increase but will tend to plateau when the annual degradation equals the annual application. Under this hypothesis the paraquat residues would follow a curved relation with time still passing through zero at time zero. This curvature must, however, be of decreasing slope (a concave function). This can be translated into hypotheses for statistical tests. This will involve testing whether the fit of the data is improved by the addition of a negative quadratic coefficient (-c. Time^2). This will be a one-sided significance test.

If a curvature is shown, then a more appropriate model would normally be one which assumes first order exponential decay between applications. To fit such a model requires allowance for the regular additional applications. For two applications the model is written as follows:

$$\text{Residue} = A_0 \cdot \text{EXP}\ (-B.T) \qquad 0<T\leq T_1$$

$$\text{Residue} = [A_1 + A_o \cdot \text{EXP}\ (-B.T_1)] \cdot \text{EXP}\ [-B.\ (T-T_1)] \qquad T>T_1$$

$$= A_1' \cdot \text{EXP}\ (-B.T')$$

Where A_o is the amount at time 0
A_1 is the addition at time T_1
T is time

The extension of this model to multiple applications is straightforward. The values A_o and A_1 represent the amount of paraquat reaching the soil at zero time and time T_1. The values used in the model could be; the amount actual applied (this assumes 100% reaches the soil), a fixed, stated fraction of the amount applied, or a fixed, but estimated from the data, fraction of the amount applied. This in the above equations would be written:

$$\text{Residue} = F \cdot A_o \cdot \text{EXP}\ (-B.T) \qquad 0\ T\ T_1$$

$$\text{Residue} = [F \cdot A_1 + F \cdot A_o \cdot \text{EXP}\ (-B.T_1)] \cdot \text{EXP}\ [-B \cdot (T-T_1)] \qquad T\ \ T_1$$

Where A_o is now the amount applied at time 0
A_1 is the amount applied at time T_1
T is time
and F is the fraction of the paraquat applied which reaches the soil.

This model can be fitted by fixing F at any particular value or by allowing F to be estimated from the data. This again is straightforward to extend to multiple applications though the actual model would look very complicated.

The linear and quadratic regression analysis was carried out using the GLM procedure in SAS (reference 4) with the NOINT (no intercept) option. Note that for PQ4 time zero is 1969. The segmented exponential curves above were fitted using the NLIN procedure in SAS. In this latter analysis, the soil samples were assumed to have been taken exactly on the anniversary of the first application. Subsequent applications were assumed to take place at times 0.01, 0.15, 0.27 and 0.54 years after that anniversary. This differs slightly from the actual times given in Appendix 2 but it is not believed that this will lead to any important inaccuracies.

4 RESULTS

4.1 Test of Curvature

Linear and quadratic equations were fitted to the data as stated earlier. The equations obtained are given in table 1. Also given in this table are the residual variances after fitting the equations. The need for the additional term, the quadratic term, in the model was tested in the standard way. The sums of squares explained by the additional term were divided by the residual variance obtained with the quadratic equation. This ratio was compared with the F-distribution with the appropriate degrees of freedom. Since the 'alternative hypothesis' is specific about the type of curvature, that is the sign of the quadratic coefficient, this could legitimately be considered as a one-sided significance test. Table 1 gives the probabilities associated with these significance tests, the two sided value is in brackets.

These results give very clear evidence of curvature in the data. The data are not therefore consistent with an hypothesis of a constant annual increment. They are therefore consistent with an hypothesis of degradation occurring.

4.2 Estimating Half Life or Degradation Rates

It is therefore appropriate to fit a biologically more meaningful model than a quadratic to the data. The model chosen was detailed earlier. The results obtained are given in Table 2. Three variations of the model have been fitted, namely

a) F = 1
b) F = 0.8
c) F estimated

Clearly any of the variations is a better fit to the data than the linear relation considered earlier. In all, the fit is better than or at least comparable with the quadratic relation. The results for variation 'a' are shown plotted in figures 1-4.

The values obtained for F, in the third variation, are all greater than 1. However, all are poorly determined, they have large 95% confidence limits, and in only one case, PQ2, is the fit improved compared to assuming F =1. Smaller values of F lead to a deterioration in fit. The estimated half lives are extremely correlated with the value of F, so that as F increases the half life estimated reduces and vice versa. The best fit to the data allowing F to vary therefore gives the lowest half lives of those considered. However, given the values of F which lead to these results - unless we are to believe that annual applications were consistently in excess of planned application - these half lives would not be put forward as realistic estimates. The other values obtained for half life in table 2 are likely to be more realistic. Taking F=1, the half life is therefore likely to be in the region of 6-7 years.

5 DISCUSSION

The analysis results show conclusive evidence that the paraquat soil residues have degraded with time and hence that the data are not consistent with an hypothesis of no degradation. Curves consistent with such degradation have therefore been fitted.

These curves are not well determined in terms of distinguishing between the proportion lost prior to soil adsorption and the proportion lost by degradation in the soil. However, one aspect of practical importance is the level of soil residue which can result over a long period of time. The most exteme situation conceivable from the results in table 2 in this respect is a half life of 14.8 years with F=0.8 (PQ2). This leads to a plateau level of 17.5 times the annual application. The extreme values for F=1 suggest a maximum of about 13 times the annual application which is probably more realistic in relation to the results observed.

6 REFERENCES

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4. 1979 SAS Users Guide. Published by SAS Institute of North Carolina.

FIGURE 1 Paraquat Concentrations in the Soil for PQ1

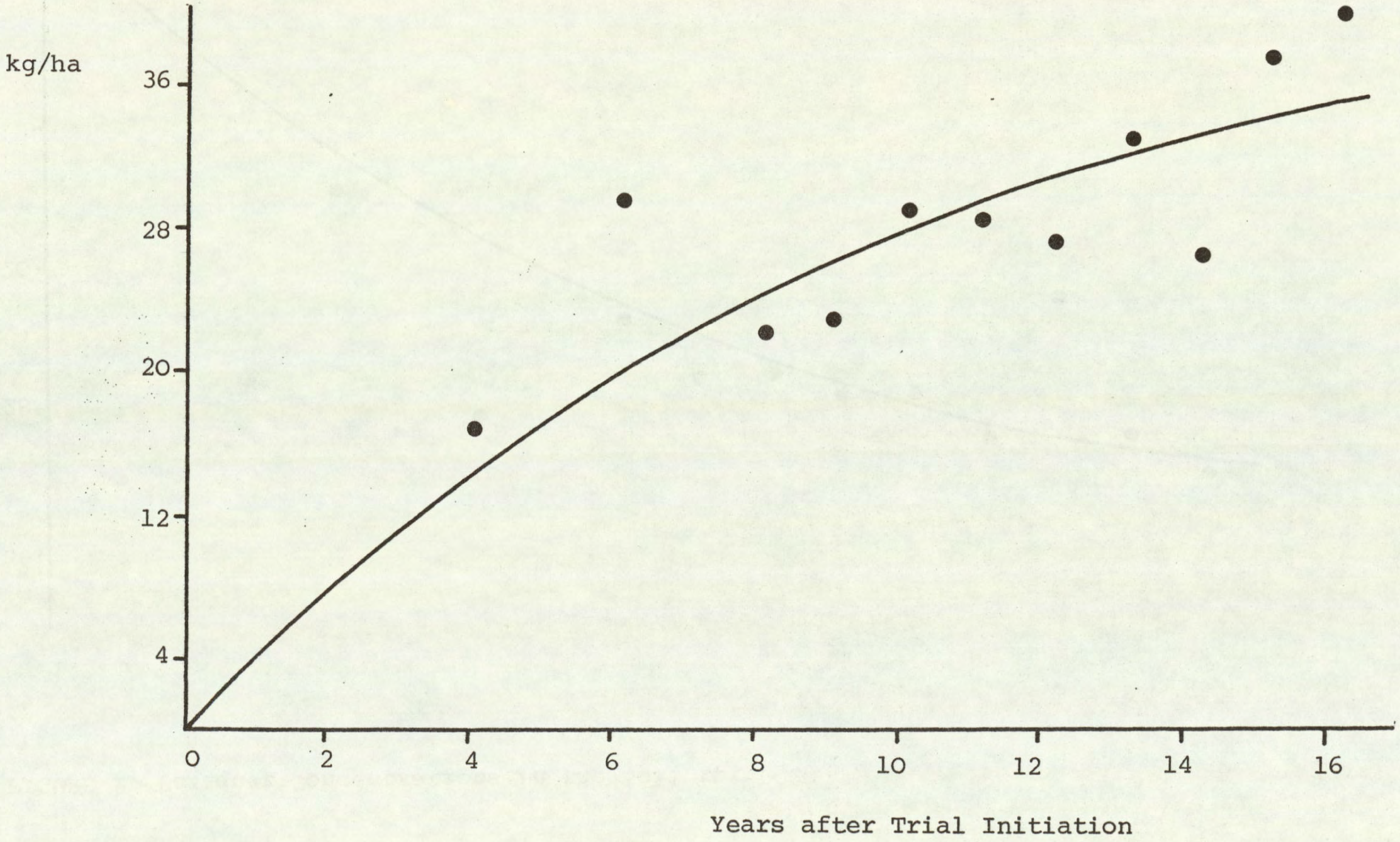

FIGURE 2 Paraquat Concentrations in the Soil for PQ2

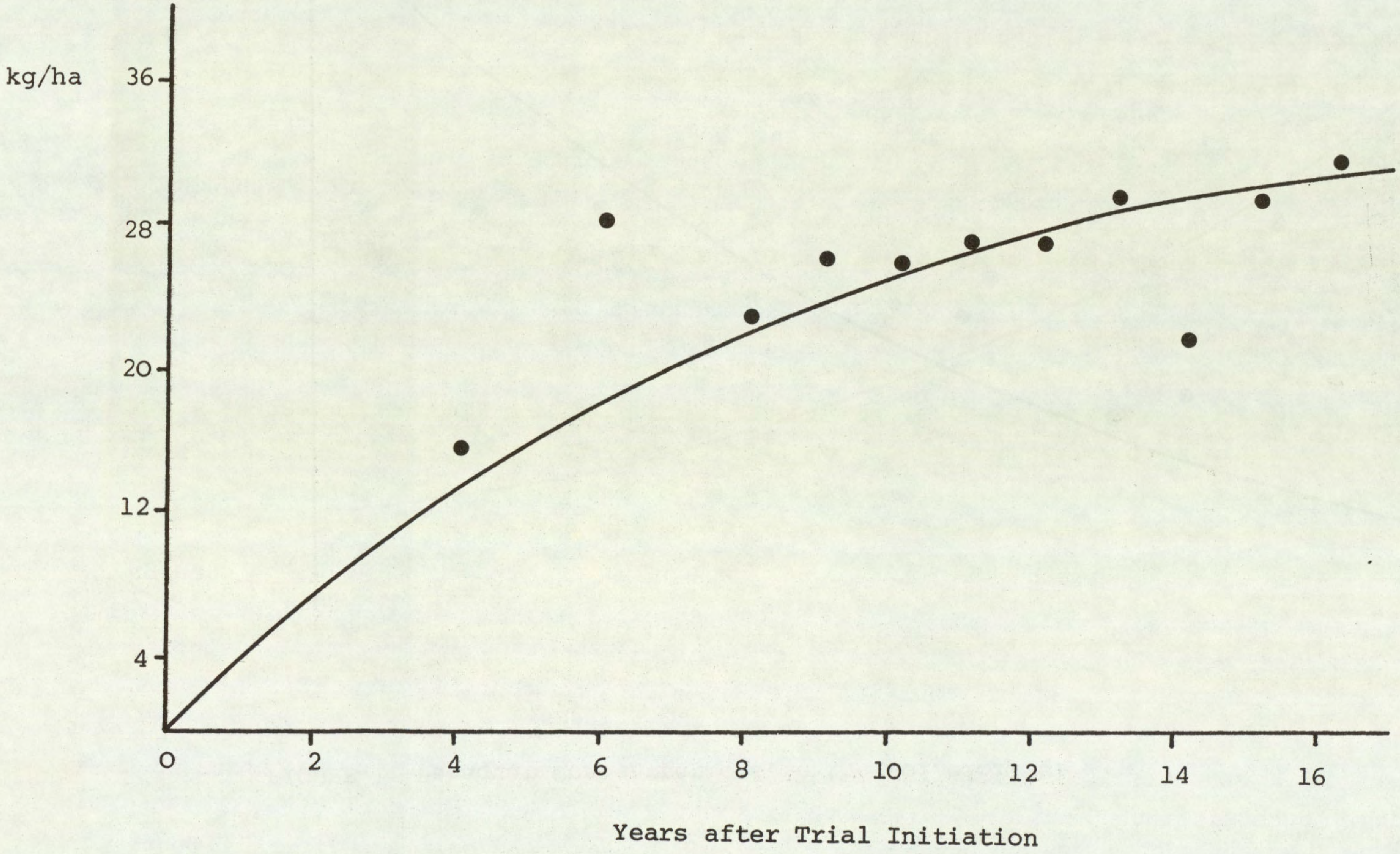

FIGURE 3 Paraquat Concentrations in the Soil for PQ3

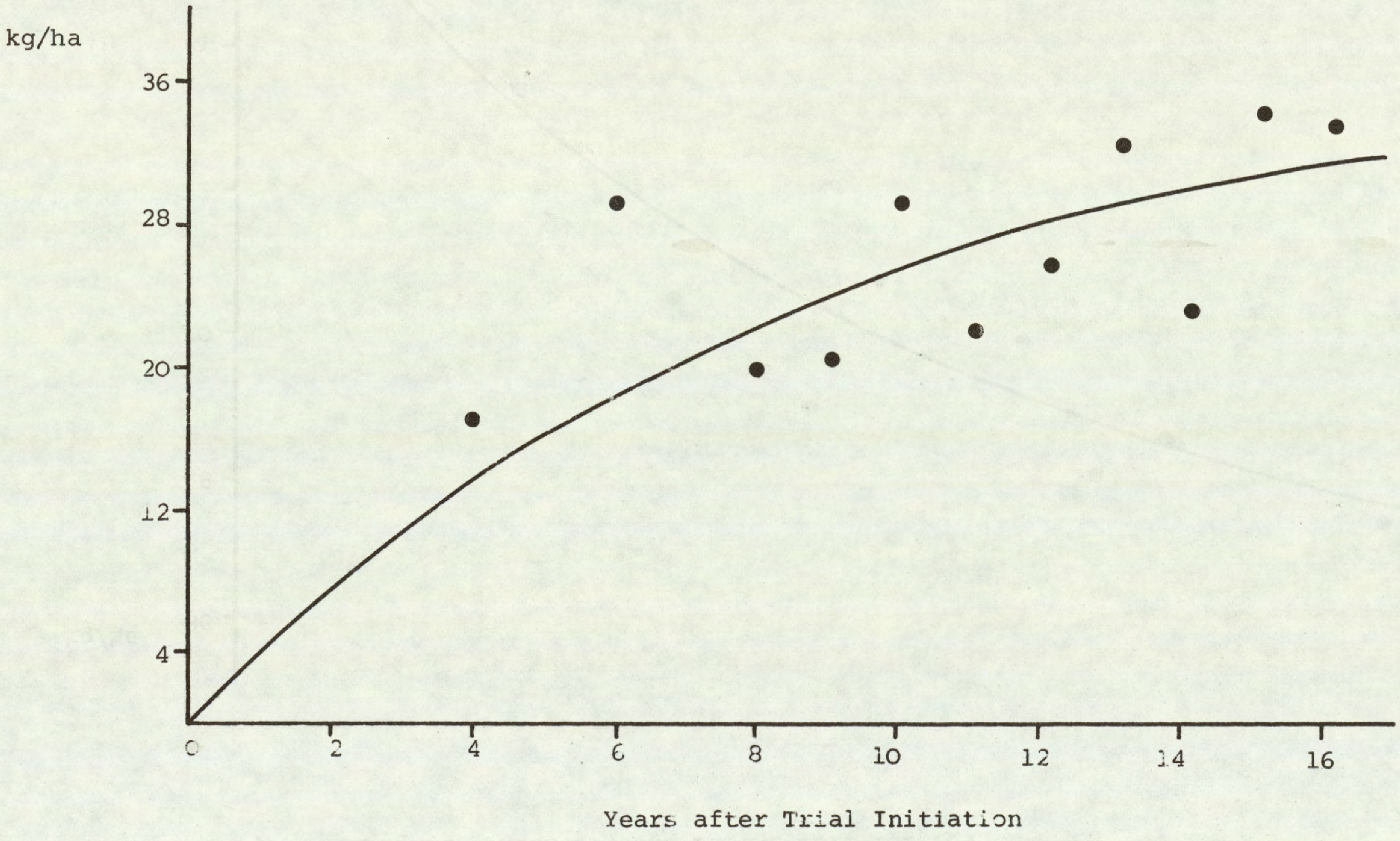

FIGURE 4 Paraquat Concentrations in the Soil for PQ4

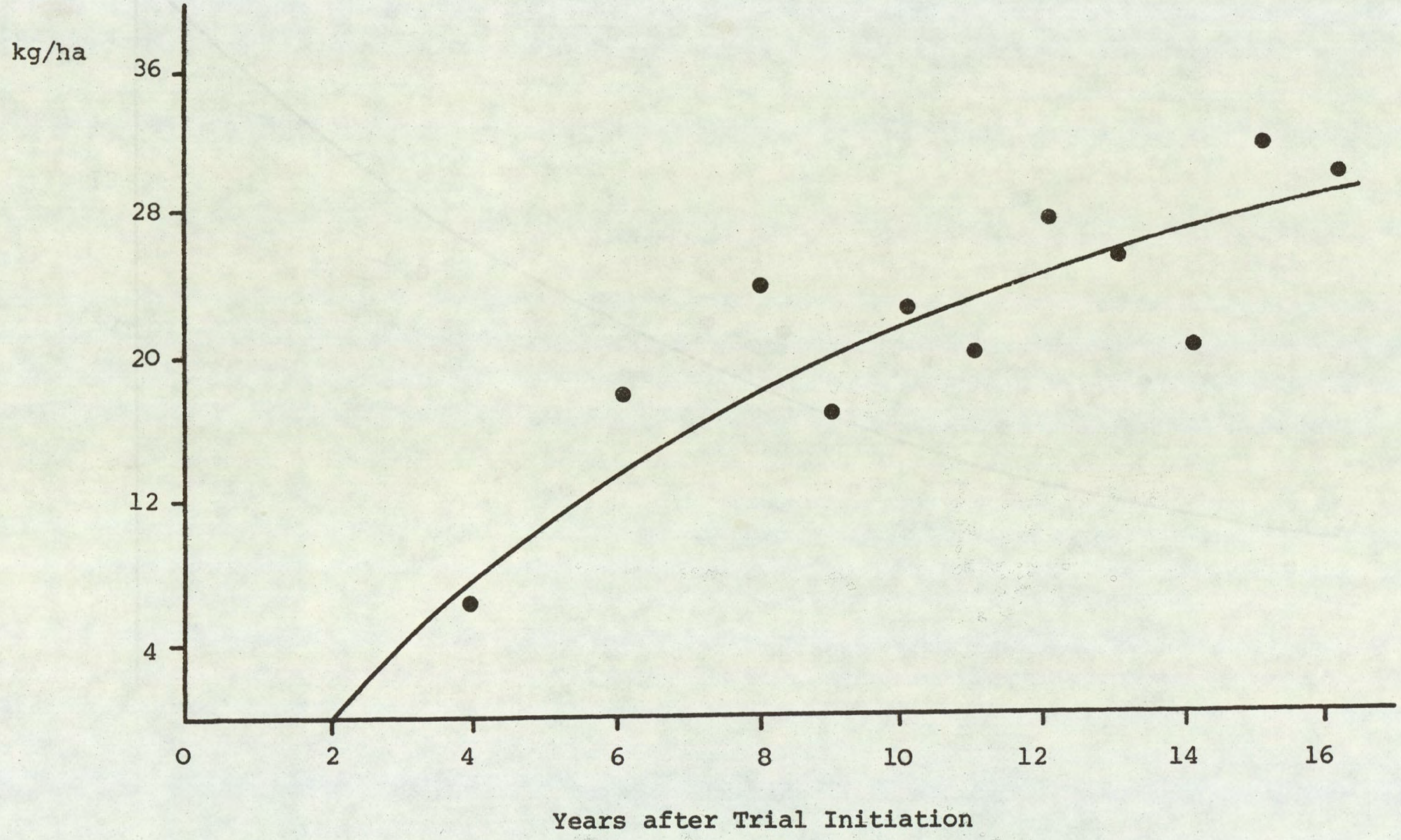

TABLE 1 RESULTS OF LINEAR AND QUADRATIC REGRESSION ANALYSIS

		PQ1	PQ2	PQ3	PQ4
Linear	Coefficient	2.46	2.20	2.25	2.36
	Residual Variance	34.3	44.8	42.7	26.4
Quadratic	Linear Coefficient	3.82	4.37	3.85	3.85
	Quad. Coefficient	-0.106	-0.170	-0.125	-0.135
	Residual Variance	24.5	15.2	30.2	16.4
	Significance Level[1] For Quad. Term	2.6% (5.2%)	0.07% (0.14%)	2.1% (4.2%)	1.3% (2.6%)

NB The Analysis was against time in years.

[1] The values in brackets apply for a two-sided significance test.

TABLE 2 RESULTS OF FITTING EXPONENTIAL MODEL

	F Fixed at 1.0		F Fixed at 0.8		F Estimated	
PQ1						
F	1.0		0.8		1.2	0.4 to 1.9
Half Life years	6.8	5.4 to 8.1	11.2	7.5 to 14.8	5.3	0.1 to 10.5
Residual Variance	19.7		22.6		21.4	
PQ2						
F	1.0		0.8		1.8	0.6 to 3.0
Half Life	5.8	4.8 to 6.8	8.7	6.2 to 11.2	2.7	0.8 to 4.6
Residual Variance	16.1		22.3		11.1	
PQ3						
F	1.0		0.8		1.4	0.3 to 2.5
Half Life	5.8	4.5 to 7.1	8.9	6.0 to 11.8	3.6	0 to 7.4
Residual Variance	24.7		28.4		25.5	
PQ4						
F	1.0		0.8		1.2	0.4 to 1.9
Half Life	5.7	4.6 to 6.9	9.0	6.3 to 11.8	4.4	0.3 to 8.4
Residual Variance	13.7		16.1		14.7	

APPENDIX 1 SOIL RESIDUES (kg/ha in 36cms)

		Sampled May/June before first application that year										
Treatment	Plot o.	1971	1973	1975	1976	1977	1978	1979	1980	1981	1982	1983
Pq 1	4	-	31.8	20.8	25.5	22.8	28.2	24.9	33.0	26.9	40.2	38.9
Pq 1	5	-	23.2	18.4	20.7	31.6	28.8	29.3	34.4	27.1	36.0	39.0
Pq 1	12	-	31.6	25.8	20.3	30.4	25.8	25.0	29.4	23.9	33.6	38.5
	Mean	16.5	28.87	21.67	22.17	28.27	27.6	26.40	32.27	25.97	36.60	38.80
Pq 2	2	-	24.4	24.0	26.2	24.4	25.7	30.7	29.4	21.3	30.7	31.7
Pq 2	7	-	31.9	23.4	23.4	24.3	32.2	18.6	25.3	20.7	28.6	33.1
Pq 2	10	-	26.4	19.8	27.3	27.1	21.4	29.8	32.8	21.4	27.4	28.9
	Mean	15.3	27.57	22.40	25.63	25.27	26.43	26.37	29.17	21.13	28.90	31.23
Pq 3	1	-	29.9	20.4	15.5	35.6	20.0	24.9	32.0	24.2	28.4	33.0
Pq 3	8	-	30.5	16.7	20.4	21.8	24.1	23.0	29.8	20.9	33.9	33.0
Pq 3	11	-	26.3	22.0	24.7	27.8	21.3	27.7	34.2	23.6	38.8	33.2
	Mean	17.0	28.90	19.70	20.20	28.40	21.80	25.20	32.00	22.90	33.70	33.07
Pq 4	3	-	17.5	13.3	19.9	21.6	15.1	24.8	26.6	20.1	32.4	30.8
Pq 4	6	-	18.2	14.9	16.7	20.1	23.3	27.6	23.8	21.3	30.6	28.6
Pq 4	9	-	-	14.4	13.7	25.2	20.8	27.9	24.6	19.5	31.3	29.6
	Mean	6.3	17.85	23.60	16.77	22.30	19.73	26.77	25.00	20.30	31.43	29.67

APPENDIX 2 PARAQUAT APPLICATION DATES

	First	Second	Third	Fourth
1967	27.4 (0.0)	6.7 (0.19)	18.9 (0.39)	16.11 (0.56)
1968	22.4 (0.99)	5.9 (1.36)	9.12 (1.62)	9.12 (1.62)
1969	14.7 (2.21)	14.7 (2.21)	8.9 (2.37)	8.12 (2.62)
1970	21.6 (3.15)	30.7 (3.26)	15.9 (3.39)	26.11 (3.58)
1971	29.6 (4.17)	16.8 (4.30)	10.9 (4.37)	2.11 (4.52)
1972	16.6 (5.14)	24.7 (5.22)	14.8 (5.28)	2.11 (5.52)
1973	14.5 (6.02)	9.7 (6.20)	4.9 (6.37)	20.11 (6.57)
1974	7.5 (7.03)	13.7 (7.21)	11.9 (7.38)	6.11 (7.53)
1975	14.5 (8.02)	30.7 (8.26)	19.9 (8.40)	14.11 (8.55)
1976	29.4 (9.01)	29.6 (9.17)	17.8 (9.31)	12.11 (9.45)
1977	2.6 (10.10)	15.7 (10.22)	7.9 (10.36)	18.10 (10.48)
1978	30.3 (10.92)	31.5 (11.09)	24.8 (11.33)	12.10 (11.46)
1979	30.5 (12.09)	2.7 (12.18)	20.8 (12.32)	15.10 (12.47)
1980	9.5 (13.03)	25.6 (13.16)	7.8 (13.28)	30.10 (13.51)
1981	5.5 (14.02)	28.7 (14.25)	3.9 (14.35)	4.11 (14.52)
1982	26.5 (15.08)	2.7 (15.18)	1.9 (15.35)	29.11 (15.59)

Figures in brackets are the times in years since the first application in 1967.

ABBREVIATIONS

ångström	Å
Abstract	Abs.
acid equivalent*	a.e.
acre	ac
active ingredient*	a.i.
approximately equal to*	≃
aqueous concentrate	a.c.
bibliography	bibl.
boiling point	b.p.
bushel	bu
centigrade	C
centimetre*	cm
concentrated	concd
concentration	concn
concentration x time product	ct
concentration required to kill 50% test animals	LC50
cubic centimetre*	cm^3
cubic foot*	ft^3
cubic inch*	in^3
cubic metre*	m^3
cubic yard*	yd^3
cultivar(s)	cv.
curie*	Ci
degree Celsius*	°C
degree centigrade	°C
degree Fahrenheit*	°F
diameter	diam.
diameter at breast height	d.b.h.
divided by*	÷ or /
dry matter	d.m.
emulsifiable concentrate	e.c.
equal to*	=
fluid	fl.
foot	ft
freezing point	f.p.
from summary	F.s.
gallon	gal
gallons per hour	gal/h
gallons per acre	gal/ac
gas liquid chromatography	GLC
gramme	g
hectare	ha
hectokilogram	hkg
high volume	HV
horse power	hp
hour	h
hundredweight*	cwt
hydrogen ion concentration*	pH
inch	in.
infra red	i.r.
kilogramme	kg
kilo (x10^3)	k
less than	<
litre	l.
low volume	LV
maximum	max.
median lethal dose	LD50
medium volume	MV
melting point	m.p.
metre	m
micro (x10^{-6})	μ
microgramme*	μg
micromicro (pico: x10^{-12})*	μμ
micrometre (micron)*	μm (or μ)
micron (micrometre)*†	μm (or μ)
miles per hour*	mile/h
milli (x10^{-3})	m
milliequivalent*	m.equiv.
milligramme	mg
millilitre	ml

† The name micrometre is preferred to micron and μm is preferred to μ.

millimetre*	mm
millimicro* (nano: $x10^{-9}$)	n or mµ
minimum	min.
minus	-
minute	min
molar concentration*	M (small cap)
molecule, molecular	mol.
more than	>
multiplied by*	x
normal concentration*	N (small cap)
not dated	n.d.
oil miscible concentrate	o.m.c. (tables only)
organic matter	o.m.
ounce	oz
ounces per gallon	oz/gal
page	p.
pages	pp.
parts per million	ppm
parts per million by volume	ppmv
parts per million by weight	ppmw
percent(age)	%
pico (micromicro: $x10^{-12}$)	p or µµ
pint	pint
pints per acre	pints/ac
plus or minus*	±
post-emergence	post-em
pound	lb
pound per acre*	lb/ac
pounds per minute	lb/min
pound per square inch*	lb/in^2
powder for dry application	p. (tables only)
power take off	p.t.o.
precipitate (noun)	ppt.

pre-emergence	pre-em.
quart	quart
relative humidity	r.h.
revolution per minute*	rev/min
second	s
soluble concentrate	s.c.
soluble powder	s.p.
solution	soln
species (singular)	sp.
species (plural)	spp.
specific gravity	sp. gr.
square foot*	ft^2
square inch	in^2
square metre*	m^2
square root of*	$\sqrt{\ }$
sub-species*	ssp.
summary	s.
temperature	temp.
ton	ton
tonne	t
ultra-low volume	ULV
ultra violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
<u>varietas</u>	var.
volt	V
volume	vol.
volume per volume	v/v
water soluble powder	w.s.p. (tables only)
watt	W
weight	wt
weight per volume*	w/v
weight per weight*	w/w
wettable powder	w.p.
yard	yd
yards per minute	yd/min

* Those marked * should normally be used in the text as well as in tables etc.

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42. The activity and post-emergence selectivity of some recently developed herbicides: KUE 2079A, HOE 29152, RH 2915, Triclopyr and Dowco 290. March 1977. W G Richardson and C Parker. Price - £3.50

43. The activity and pre-emergence selectivity of some recently developed herbicides: dimefuron, hexazinone, trifop-methyl, fluothiuron, buthidazole and butam. November 1977. W G Richardson and C Parker. Price - £3.75.

44. The activity and selectivity of the herbicides: ethofumesate, RU 12709 and isoproturon. December 1977. W G Richardson, C Parker, & M L Dean. Price - £4.00

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62. The activity and pre-emergence selectivity of some recently developed herbicides: UBI S-734, SSH-43, ARD 34/02 (= NP 55), PP 009 and DPX 4189. February 1981. W G Richardson, T M West and C Parker. Price - £3.50

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