

PROTOCOL TO TEST POSSIBLE CROSS-CONTAMINATION OF SAMPLES DURING SOIL CORING PROCEDURES - SAMPLES TAKEN 6/18/86

I. Objective; To determine whether or not cross-contamination of samples occurs during soil coring techniques. Two techniques will be tested: 1. The technique used for the Mobile Drill soil coring apparatus, and 2. The E.P.A. methodology for obtaining soil cores by bucket augering techniques.

II. Personnel; Don Weaver will supervise the study. John Troiano will be responsible for study design and data analysis and Cindy Garretson will be responsible for site preparation.

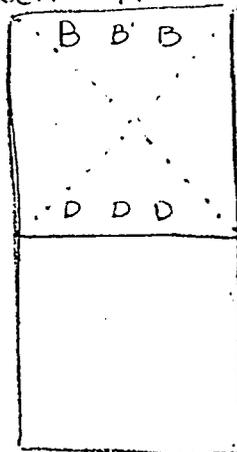
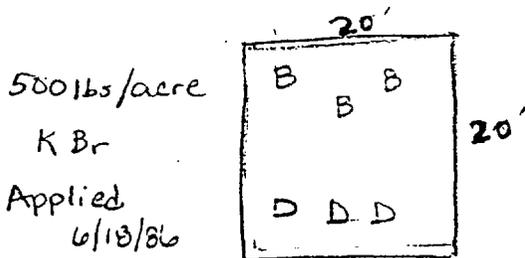
III. Study Design; Cross-contamination of samples will be tested by applying a tracer to the surface of soil immediately before obtaining soil cores. The plot will be located at the Fresno State college test site adjacent the irrigation test site. The plot size will be 20 feet by 20 feet. Six soil cores to 10 feet will be taken for each sampling method. Soil samples within each core will be 6 inches in depth. The cores will be immediately packaged and delivered to the laboratory. The coring will occur in mid-June.

Since the site at Fresno has not received recent applications of agricultural chemicals, potassium ~~chloride~~ bromide will be used as the tracer applied at a rate of 500 lbs/acre.

Potassium Bromide applied morning of 6/18/86. (Dissolved in water and sprayed on with Hudson sprayer)

IV. Laboratory Analyses; Laboratory analyses will be conducted by APPL labs in Fresno. Approximately 240 6-inch cores will be generated. The standard quality assurance and quality control procedures used by APPL labs will be followed. At a cost of approximately \$30 per analysis, the total cost will be \$7200.

Half the samples were taken from the 20' x 20' plot  
Half the samples were taken from the old winter rain plot in 4 sections where KBr had been applied.



150 lbs/acre KBr  
Applied  
12/4/85 before  
bulk of winter rain

0 - bucket auger  
1 - mobile drill

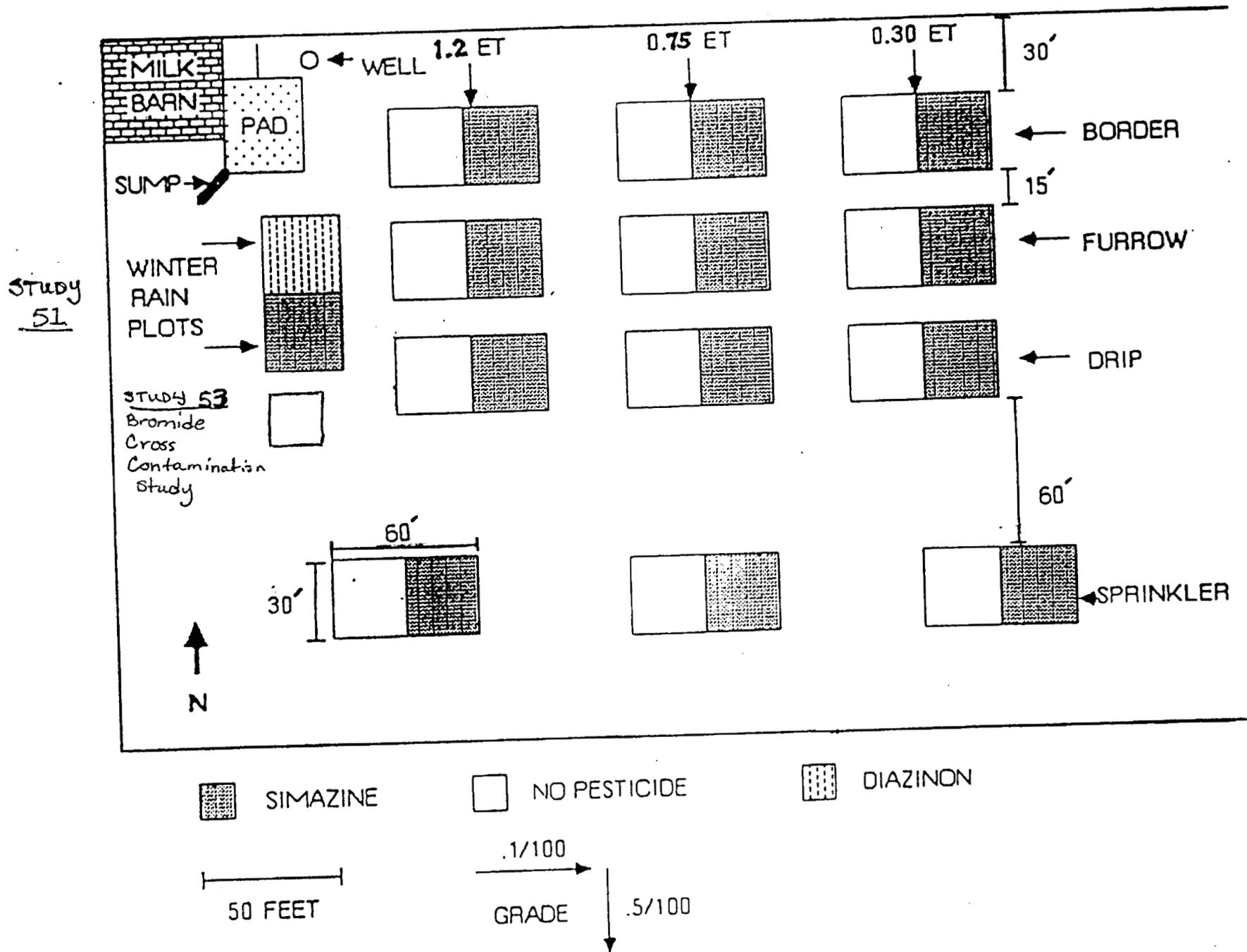
Study #53

6/18/86

$$20 \times 20 = 400 \div 43,560 = 0.00918 \text{ acres} \times 150 \text{ lb/ac} = 1.377 \text{ lbs} \times 454 \text{ g} = 625.3 \text{ g}$$

Applied 625.3 g KBr to plot on morning of 6/18/86  
It was dissolved in distilled water and applied  
to plot with hand sprayer.

Figure 1. Positioning of plots for irrigation study at Fresno field facility.



**Bromide electrode** The Orion model 94-35 bromide ion-selective electrode is a solid state device that measures bromide ion in water and in some organic solvents. It must be used with a suitable reference electrode.

The electrode's sensor is a silver bromide/silver sulfide membrane bonded into an epoxy body (See Fig. B-2). When this membrane is in contact with a solution, a potential dependent upon the level of bromide ion in solution develops across the membrane. The potential is measured against a reference electrode.

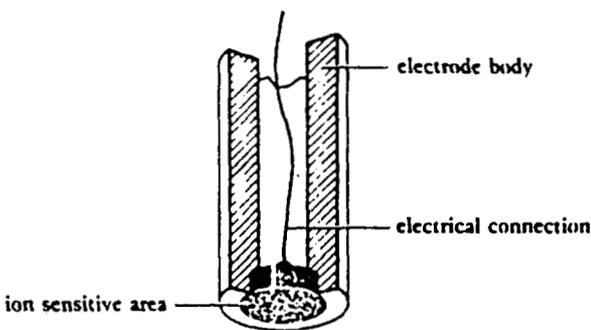


Fig. B-2: Construction of the bromide electrode.

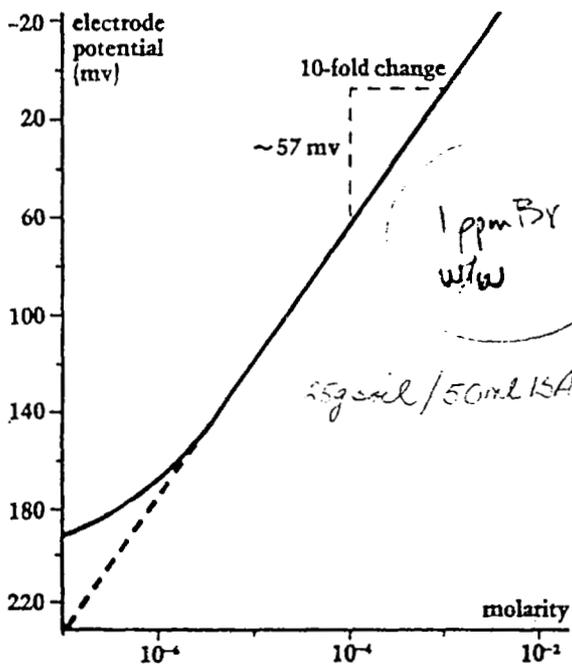


Fig. B-3: Typical bromide electrode calibration curve.

The bromide electrode should not be used in solutions containing ions which form insoluble salts with silver, since these will react with the surface of the membrane, causing the electrode to malfunction. In addition, strong reducing agents should be avoided as they will react to form a layer of silver on the membrane. In either case, performance is restored by polishing the membrane surface.

Sulfide and cyanide, which form insoluble silver salts, can be removed from samples with nickel (II) nitrate. Measurements can be made in solution containing oxidizing agents such as copper (II), iron (III), and permanganate ions, or in solutions containing chloride. Mercury should be absent from samples.

The bromide electrode can be used at temperatures ranging from 0 to 100°C and at pH from 0-14. Bromide concentrations from  $5 \times 10^{-6}$ M to 1.0M can be measured directly (See Fig. B-3).

**IM9435, Model 94-35 Bromide Electrode Instruction Manual.** Orion Research.

**B799, "Determination of Inorganic Bromide in Soils and Plant Tissues with a Bromide Selective-Ion Electrode."** Abdalla, N.A.; Lear, B. *Comm. in Soil Sci. and Plant Anal.*, 1975, 6(5), 498.

**B128, "Specific-Ion Electrode Measurements on Br<sup>-</sup>, Cl<sup>-</sup> and F<sup>-</sup> in Atmospheric Precipitation."** Harriss, R.C.; Williams, H.H. *J. Appl. Meteorol.*, 1969, 8(2), 229.

**B716, "Use of Bromide Electrodes for Rapid Screening of Elevated Bromide Concentrations in Biological Fluids."** Poser, S.; et al., *Z. Klin. Chem. Klin. Biochem.*, 1974, 12, 350.

**B891, "Determination of Bromide Ion Content of Cereals and Other Foodstuffs."** Banks, H.J.; Desmarchelier, J.M.; Elek, J.A. *Pestic. Sci.*, 1976, 7, 595.

**Bromide in soils and plant tissues** Bromide is measured in soils and plants to assess contamination resulting from brominated pesticides.

**Analysis:** Bromide is measured using the 94-35 bromide electrode and 90-02 reference electrode. Standards which bracket the expected bromide concentration are prepared by diluting 0.1M NaBr extraction (No. 943506).

Samples are prepared with distilled water. The samples are stirred for 30 minutes and filtered. Samples and standards are adjusted to a constant ionic strength by adding 5M NaNO<sub>3</sub> (No. 940011). Sample values are read either directly, using a model 901 microprocessor analyzer, or from a calibration curve.

**\*B799, "Determination of Inorganic Bromide in Soils and Plant Tissues."** Abdalla, N.A.; Lear, B. *Comm. in Soil Sci. and Plant Anal.*, 1975, 6(5), 498.

**B891, "Determination of Bromide Ion Content of Cereals and Other Foodstuffs."** Banks, H.J.; Desmarchelier, J.M.; Elek, J.M. *Pestic. Sci.*, 1976, 7, 595.

**Bromide in wine** High levels of bromide are considered an impurity in wine.

**Analysis:** Bromide is measured with the 94-35 bromide electrode and 90-01 reference electrode using the known addition technique.

Samples are prepared by adding 3.75M phosphoric acid, saturated KNO<sub>3</sub>, and 1M cupric sulfate. To each 100 ml of sample is added 1 ml of a bromide standard prepared from 0.1M NaBr (No. 943506). Sample concentration is determined from the change in potential after addition of standard. Direct concentration readout can be obtained using the 901 microprocessor analyzer.

**B821, "Alcoholic Beverages: Analysis of Bromide Ion in Wine by Ion Selective Electrode."** Graf, J.E.; Vaughn, T.E.; Kipp, W.H. *JAOAC*, 1976, 59(1), 53.