sample extractives with compensation for the partitioning ratios of these metabolites in benzene-water systems.

The results obtained from the various stages of cattle feed processing are collated in Table II. Comparison of the residues in unwashed peel with those in the ground peel shows the loss of Bidrin from the detergent wash of the 15- and 30-day samples to be 42 and 28%, respectively.

The ground peel contained 75 to 80% water as compared to 7.0 to 11.5% in finished cow feed. If no Bidrin were lost after the ground peel stage, the residues in the cow feed from the 15- and 30-day samples would average 16.8 and 10.7 p.p.m., respectively. However, the 15-day sample lost 80%, while the 30-day sample lost only 60% of the Bidrin present in the ground peel stage. Possible explanation of this apparent discrepancy is the higher final moisture content of the 30-day samples; the

laboratory processing procedure is analytically reproducible. Thus, there may be a critical moisture content above which very little Bidrin is lost but below which Bidrin is lost very rapidly. However, it is clear that laboratory processing of orange fruits into citrus "pulp" cattle feed does eliminate a significant amount of any Bidrin residue which may be present initially.

There was no detectable ($\sim$0.1 p.p.m.) storage (5° C.) deterioration of fortified control peel, juice, and citrus pulp cattle feed over the 4-month duration of this program.

The winter application on mature navel oranges (Table II) afforded significantly greater peel residues than the same dosage in a summer application on mature Valencia oranges.

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HERBICIDE SAMPLING APPARATUS

Collection Technique for Aerosol and Gaseous Herbicides

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An air-sampling system for the differential collection of aerosol and gaseous fractions of airborne herbicides consists of a rotating disk impactor for collecting aerosol droplets down to approximately 3 microns in diameter, followed by a midget impinger to collect the gaseous fraction. The impactor was specially designed and constructed of glass, Teflon, and stainless steel to prevent contamination of the collection fluid with substances that interfere with electron capture gas chromatography. Incoming air impinges on the impaction disk, which slowly rotates through a fluid well containing n-decane. The impacted droplets wash off into the collection fluid. The disk then passes through a Teflon squeegee to remove the adhering droplets presenting a smooth surface containing a fluid film upon which the air stream impinges. The collection efficiency of the system for gaseous and aerosol forms of 2,4-D esters has been studied under laboratory conditions. The system was used at two field sampling sites for approximately 3 months.

I N THE EVALUATION of atmospheric drift of herbicides a method was required for continuous differential collection of aerosol and gaseous fractions. Since a modified midget impinger was used in our earlier studies of total atmospheric herbicide (1), a suitable system was needed to collect the aerosol fraction and pass the gaseous fraction to the midget impinger.

The requirements of this aerosol collection device included: continuous sampling for 24-hour periods without presence of an operator, a relatively low air sample flow rate of 1 liter per minute for compatibility with a midget impinger, a minimum collection efficiency of 90%

for aerosols in the range of 1 to 50 microns, at least 90% of the smaller droplets and gases not collected, aerosol sample in a form amenable to electron capture gas chromatographic analysis, the sample collected in 5 to 20 ml. of absorption liquid, and the complete unit portable and operable from a 12-volt d.c. source.

Aerosols can be collected by a number of methods including filtration, centrifugation, electrostatic precipitation, and impaction. Any of these techniques could be used to collect aerosols containing compounds of 2,4-D, simultaneously separating them from concomitantly occurring gases. However, the direct application of these techniques

to the collection of a 24-hour sample of aerosols containing the more volatile esters of 2,4-D would suffer from continuing loss of the collected esters to the sampled air stream during the remainder of the sampling period. A survey of existing aerosol collection devices (2, 3) indicated that no device meeting the above requirements was available; therefore, a suitable sampler was designed.

Schadt et al. (6) described a rotary, electrostatic precipitator in which sulfuric acid aerosol was deposited on a rotating, stainless steel disk which dipped into a flowing stream of water. This concept was modified to provide a rotary impactor constructed of glass and Teflon

for the collection of aerosols containing 2,4-D compounds. The primary advantage of the rotary impactor is the removal of the impacted aerosol from the air stream and into the collecting liquid. Suitable materials of construction include glass, stainless steel, and Teflon. Glass and Teflon were chosen to provide maximum visibility for observation of the functioning of the aerosol collector. The impactor should include a liquid well to minimize the volume of collecting medium required.

Based on these considerations an experimental design of a rotating disk impactor was evolved providing convenience of sample changing with minimum opportunity of sample contamination. Two field samplers were constructed, tested, and used in atmospheric herbicide studies.

Apparatus Design

Sedimentation velocities of small particles (3) indicated that particles within the range of 1 to 50 microns should be sampled for atmospheric herbicide (Table I).

Droplets larger than 50 microns in diameter would tend to sediment out so rapidly that they would be of little interest as an atmospheric contaminant, while particles smaller than 1 micron in diameter would settle so slowly that they would tend to act like gases and should be collected with the gaseous fraction.

Preliminary Design Considerations. An impactor consists of a critical orifice through which the air sample is drawn and a rotating impaction plate upon which the air jet impinges. The particles in the air stream collect on the plate because their inertia is greater than that of the air molecules. Ranz and Wong (4) defined a dimensionless inertial parameter, Σ, in terms of the characteristics of the impactor stage and the particles being collected.

The equation for Σ is:

$$\Sigma = CP_pV_oD_p^2/(18\mu D_c)$$

where P_p = density of aerosol particles, gram/cc.

V_o = velocity of aerosol jet, cm./sec.

D_p = effective diameter of aerosol particle, cm.

μ = viscosity of air, poise

D_c = diameter of round jet orifice, cm.

$C = 1.00 + (0.16 \times 10^{-4})/D_p$.

C is a dimensionless empirical correction factor for the resistance to movement of small particles in air.

Since V_o = flow rate per cross-section area of jet = $4 \times$ flow rate$/\pi D_c^2$, one may write:

$$\Sigma = 4CP_pD_p^2 \times \text{flow rate}/18\pi\mu D_c^3 \quad (1)$$

The proper jet diameter for collecting particles down to 1 micron in diameter may be calculated using Equation 1 and the following conditions:

P_p = 1.0 gram/cc. for aqueous droplets

D_p = 1 micron or 1×10^{-4} cm.

Flow rate = 1 liter/min. or 16.7 cc./sec.

μ = 1.8×10^{-4} poise

Σ = 0.38 (characteristic of a round jet)

D_c = 5.6×10^{-2} cm. or 0.56 mm.

A glass capillary tube 0.55 mm. in diameter was therefore selected for the jet.

For continuous operation it was necessary to remove the collected material from the impactor plate. This was accomplished by using a circular plate and rotating the lower portion of it into a suitable solvent. The plate was rotated by a 1-r.p.m. synchronous 120-volt, 60-cycle a.c. timer motor coupled to the plate shaft.

A Teflon wiper blade removed excess collection fluid from the disk, reducing evaporation of solvent, and facilitating the drying of the disk prior to exposure to the air jet. It also provided partial separation between the air stream and the main body of the collection fluid, thereby reducing solvent loss by direct evaporation into the air stream and minimizing absorption of gases from the air stream.

The construction and operation of the

Table I. Rate of Fall of Spherical Particles in Still Air (3)

Particle Diameter, μ	Rate of Fall, Cm./Sec.
100	30
10	0.3
1	0.003
0.1	0.00009

CORNING CUP
7 CM O.D. x 3 CM D.P.

$2\frac{1}{4}$ DIA. S.S. DISK

$\frac{1}{4}$ S.S. ROD

TEFLON WIPER BLADE

3 DIA. TEFLON IMPACTOR COVER

OUTLET # 12/5 ⚲

0.5 MM CAPILLARY

INLET & ORIFICE # 12/5 ⚲

DRIVE COUPLING

SUPPORT PLATE

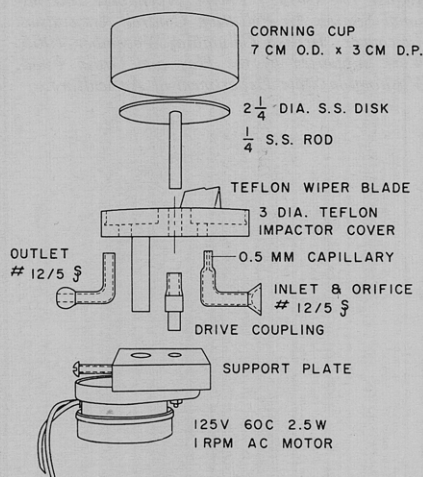125V 60C 2.5W 1 RPM AC MOTOR

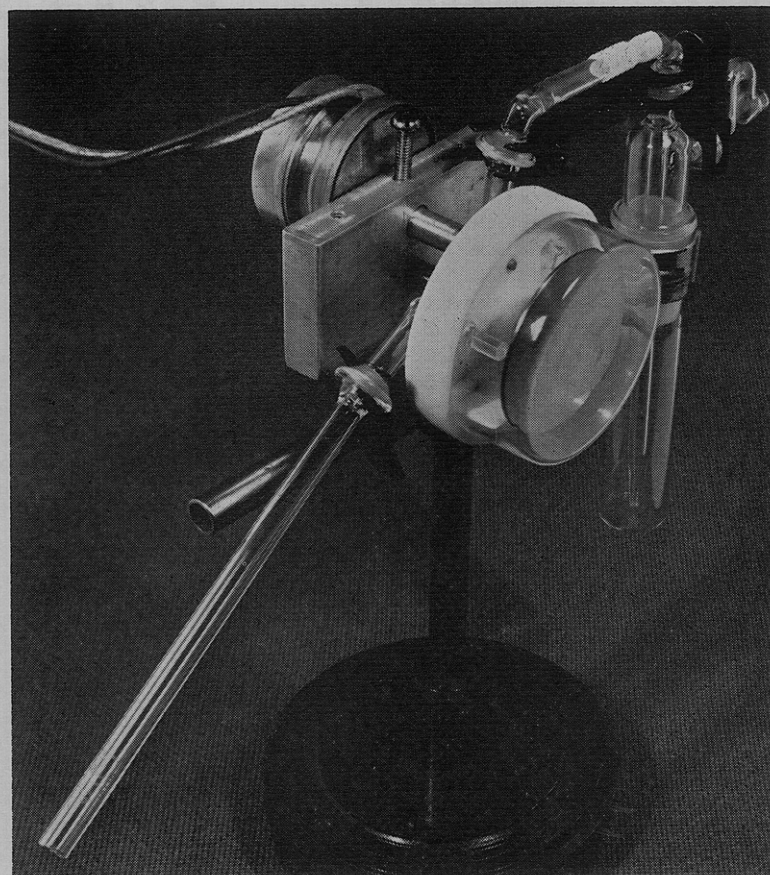Figure 1. The rotating disk impactor, disassembled

Figure 2. The rotating disk impactor coupled with a modified midget impinger as a differential collection system

rotating disk impactor (RDI) were simplified by mounting the wiper blade, stainless steel disk, glass capillary inlet jet, glass outlet tube, and metal support rod on a 3-inch-diameter Teflon lid. The distance between the top of the jet and the disk was set at 1.5 mm. The glass solvent cup (made from a Pyrex No. 3140 evaporating dish cut to a 3-cm. depth and fire polished) was attached to the lid by means of an air-tight, press-fit seal. This entire assembly was attached to the rotating motor and base plate by means of a setscrew seating on the support rod.

Sample changing was simplified by use of 12/5 ₷ glass ball and socket joints on the outlet and inlet, respectively. The final design of the rotating disk impactor is shown in Figures 1 and 2.

Apparatus Tests and Results

Equipment and Procedure. The sampling system used to determine the collection efficiency of the RDI has been described (7). The RDI was placed ahead of the midget impinger in the air sample stream, in the cold air space of the water bath to maintain a uniform operating temperature.

In operation, the RDI disk is in a vertical position. When the sample is changed, the RDI is detached from the sample stream and rotated so that the disk is horizontal. The lid is then removed from the solvent cup, the disk is rinsed, and the lid is attached to another solvent cup containing a new aliquot of collection fluid. The RDI is then returned to the sampling system for further sampling.

The collection efficiency of the RDI for gases and for aerosols was determined by comparing the total amount of material collected in the RDI and the midget impinger. Gas dilutions of isopropyl and butyl 2,4-D esters were prepared by placing open containers of the pure esters near the sample inlet. The collected samples were analyzed by electron capture gas chromatography (7).

Aerosols were prepared with a De-Vilbiss No. 40 glass nebulizer containing a 1% solution of ester in n-decane. The particle size distribution produced by the nebulizer was obtained by spraying dye-containing aerosol into a closed chamber and allowing sedimentation of the droplets onto glass slides. The slides were then qualitatively examined with a microscope having a calibrated eyepiece. The aerosol consisted of droplets 1 to 10 microns in diameter with the greater numbers falling in the intermediate diameters.

Collection Efficiency Tests. The gas collection efficiency of the midget impinger has been studied by Roberts and McKee (5). They reported a collection efficiency of 85 to 95% when the air sampling rate was 1 to 4 liters per minute, the collected material was readily wetted by or soluble in the collection fluid, and the concentration of the collected material in the collection fluid did not approach saturation. This study substantiates this value for both gases and aerosols when 2,4-D esters are collected in n-decane at a flow rate of 1 liter per minute (Table II).

Table II. Collection Efficiency Ranges for 2,4-D Type Herbicides

| | | Efficiency, % | |
Form	Atm. Concn. Range, µg./Cu. M.	Midget impinger	Rotating disk impactor
Gaseous	0–100		
Isopropyl 2,4-D		97.0	26.3
Butyl 2,4-D		97.0	17.1
Aerosol (1–10µ)	0–200		
Isopropyl 2,4-D		89.0	62.1
Butyl 2,4-D		85.8	71.2

The midget impinger was assumed to collect all the 2,4-D esters which were not collected in the RDI. The percentage collection efficiency of the RDI was reported in terms of the amount of ester in the RDI divided by the total found in both the RDI and the midget impinger. Since the efficiency calculations are based on the total compound found in the first and second collectors,

one cannot assume an efficiency of 100% when no compound is detected in the second collector. The efficiencies were conservatively calculated by assuming that up to the detectable concentration might be present in the second collector.

Tests with gaseous 2,4-D esters indicated a variable collection efficiency for the RDI between 10 and 30%. The variable collection of gases in the RDI may be due to absorption of the more volatile gases from the air stream in the film of collection fluid remaining on the disk. The comparative amount of collected esters appeared to be related to the relative volatility of the isopropyl and butyl esters.

Tests of the RDI for collection of an n-decane aerosol containing 2,4-D esters in 1- to 10-micron droplets showed efficiencies of 60 to 75%. Efficiencies of less than 100% indicate a particle collection limit between 1 and 10 microns. The lower particle size collection limit was estimated to be between 3 and 6 microns. This compares well with the desired value of 1 micron, since the equation used to determine the jet diameter was only approximate.

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