MALEIC ANHYDRIDE

Method no.:	25
Matrix:	Air
Target concentration:	1 mg/m^3 (OSHA PEL)
Procedure:	Collection and derivatization on a sampling train consisting of two adsorption tubes connected in series. The first tube contains XAD-2 coated with *p*-anisidine and the second tube contains untreated XAD-2. The anisidine-maleic anhydride derivative is desorbed with methanol and analyzed by reverse phase HPLC with a UV detector.
Recommended air volume and sampling rate:	20 L at 0.1 L/min
Reliable quantitation limit:	0.005 mg/m^3
Standard error of estimate at the target concentration: (Section 4.7.)	7.6%
Special requirements:	*p*-Anisidine coated sorbent tubes should be shielded from sunlight at all times. The recommended sampling rate should not be exceeded.
Status of method:	An air sampling and analytical method that has been evaluated according to the criteria established by the Organic Methods Evaluation Branch.

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Chemists: Keith Motley
Tom Plummer

Carcinogen/Pesticides Branch
OSHA Analytical Laboratory
Salt Lake City Utah

1. General Discussion

1.1. Background

1.1.1. History

The procedure for determining maleic anhydride air concentrations which employs isopropanol impingers encounters a serious interference with the presence of maleic acid. The monoisopropyl ester of maleic acid was previously considered to be formed solely from the reaction of maleic anhydride with isopropanol and hence was the compound considered to exclusively represent the concentration of maleic anhydride. It can be shown, however, that maleic acid also reacts with isopropanol to form this ester in significant quantities. Further, the maleic acid itself may be either of ambient origin or formed from the hydrolysis of maleic anhydride utilizing trace amounts of water in the isopropanol impingers. In view of the myriad of equilibria and unknown factors involved, the use of isopropanol impingers for the quantitative determination of maleic anhydride is found to be lacking in both specificity and accuracy. (Section 4.8.)

During the past several months, attempts have been made at this lab to selectively derivatize maleic anhydride in the presence of maleic acid. Among the derivatizing agents tried were triphenyl phosphine, various Diels-Alder reagents such as cyclopentadiene and 1,3-diphenyl isobenzofuran, imidazoles and thiazoles. All of the above fell short of the desired goal due either to stability problems or the inability to chromatograph and analyze the resultant derivative. *p*-Anisidine, however, serves as an excellent analytical derivatizing agent for maleic anhydride due to: 1) its ability to react with the anhydride in high yield (Figure 1.1.1.), 2) the ease with which the derivative is chromatographed and, 3) the convenience of collection on solid sorbent tubes rather than liquid impingers (Ref. 5.1.).

The collection and analysis of maleic anhydride using *p*-anisidine on solid sorbent tubes was therefore examined and the results presented in the method that follows.

1.1.2. Toxicology (This section is for information only and should not be taken as the basis for OSHA policy).

Maleic anhydride is a severe irritant to the eyes, skin and respiratory tract which can, upon exposure, produce intense burning sensations in the eyes and throat with coughing and vomiting. Among workers repeatedly exposed to 5-10 mg/m^3, toxic effects included ulceration of nasal mucous membranes, chronic bronchitis, and in some cases, asthma. Other potential effects of exposure are derma titis, pulmonary edema, respiratory sensitization, skin sensitization, photophobia and double vision. (Ref. 5.2.)

1.1.3. Uses where exposure may occur

Most maleic anhydride now produced in the United States is obtained from the catalytic oxidation of either benzene or butene. An exception is the isolation of maleic anhydride as a by-product in the production of phthalic anhydride. Approximately 50% of the maleic anhydride produced is used in the manufacture of polyester resins. The remaining maleic anhydride is used in the preparation of fumaric acid, agricultural pesticides, alkyd resins and miscellaneous chemical products. No literature data indicating the number of workers potentially exposed to maleic anhydride were found. (Ref. 5.3.)

1.1.4. Physical properties

Maleic Anhydride (Ref. 5.6.)

molecular weight:	98.06
melting point:	53°C
boiling point:	202°C
vapor pressure:	1 mmHg at 44.0°C
structure:	Figure 1.1.4.
synonyms:	2,5-furandione, toxilic anhydride, *cis*-butenedioic anhydride.

Maleic Anhydride-*p*-Anisidine Derivative (experimental)

molecular weight:	221.21
melting point:	184°C
boiling point:	unknown
appearance:	yellow-green powder or crystals
structure:	Figure 1.1.4.

1.2. Limit defining parameters

1.2.1. Detection limit of the analytical procedure

The detection limit of the analytical procedure is 2.4 ng of maleic anhydride per injection. This is the amount of maleic anhydride necessary to produce a derivative peak whose height is approximately five times the baseline noise height. (Section 4.1.) The cited amount is expressed as maleic anhydride even though the derivative was the actual substance spiked.

1.2.2. Detection limit of the overall procedure

The detection limit of the overall procedure is 97 ng of maleic anhydride per sample or 0.005 mg/m^3 based on the recommended air volume of 20 L. This is the amount of maleic anhydride spiked on the sampling device which allows recovery of an amount of that derivative equivalent to the detection limit of the analytical procedure. (Section 4.2.) The cited amount is expressed as maleic anhydride even though the derivative was the actual substance spiked.

1.2.3. Reliable quantitation limit

The reliable quantitation limit is 97 ng of maleic anhydride per sample or 0.005 mg/m^3 based on the recommended air volume. This is the smallest amount of maleic anhydride which can be quantitated within the requirements of 75% recovery and 95% confidence limits of less than ±25%. (Section 4.2.) The cited amount is expressed as maleic anhydride even though the derivative was the actual substance spiked.

1.2.4. Sensitivity

The sensitivity of the analytical procedure over a concentration range representing 0.5 to 2 times the PEL based on the recommended air volume is 2019 area units/ng of maleic anhydride detected as the derivative. The sensitivity is determined by the slope of the calibration curve. (Section 4.4.) The cited amount is expressed as maleic anhydride even though the derivative was the actual substance spiked.

1.2.5. Recovery

The recovery of analyte from the sorbent tubes must be greater than 75%. The average recovery over the range of 0.5 to 2 times the PEL is 96%. (Section 4.6.)

1.2.6. Precision of the analytical method

The pooled coefficient of variation obtained from replicate determinations of analytical standards at 0.5, 1, and 2 times the PEL is 0.0095. (Section 4.3.)

1.2.7. Precision of the overall procedure

The overall procedure must provide results that are ±25% or better at the 95% confidence level. The precision at the 95% confidence level for the 15-day storage test is ±15.9%. This figure includes an additional ±5% for sampling error. (Section 4.7.)

1.3. Advantages

1.3.1. The major advantage is the quantitative recovery of maleic anhydride as the derivative without interferences from maleic acid or degradation of the highly unstable and reactive anhydride.

1.3.2. The sampling apparatus is compact, easy to use, and has no liquid spill potential.

1.3.3. The analytical procedure is straight-forward and reproducible with detection limits well below the PEL value.

1.4. Disadvantages

1.4.1. The derivatization tube contains *p*-anisidine, which is listed in some journals as a suspected carcinogen. This necessitates a backup XAD-2 tube in the sampling train to collect any *p*-anisidine lost from the first tube. *p*-Anisidine is known to collect on XAD-2 tubes (Ref. 5.4.).

1.4.2. The capacity of the treated sorbent tube is limited but should give quantitative recovery up to 4 times the PEL at the recommended air volume.

2. Sampling Procedure

2.1. Apparatus

2.1.1. XAD-2 sorbent tubes coated with 500 µg of *p*-anisidine. Instructions for the preparation of these tubes are presented in Section 4.9.

2.1.2. Untreated XAD-2 sorbent tube used as an in-series backup to the *p*-anisidine coated tube.

2.1.3. An air sampling pump with a flow rate which can be calibrated to within ±5% of the recommended 0.1-L/min flow rate while the sampler is in line.

2.2. Reagents

None required.

2.3. Sampling technique

2.3.1 The air sampling train is composed of one treated XAD-2 tube followed by an untreated XAD-2 tube in series. The tubes are connected with a modified end cap which has had the closed portion cut off. The "B" section of the treated tube should be followed by the "A" section of the untreated tube. (Figure 4.5.)

2.3.2. Connect the sampling train to the sampling pump with a piece of flexible tubing. Cover each tube with masking tape or other material to prevent exposure to sunlight.

2.3.3. The air sampler should be placed in a vertical position to minimize channeling.

2.3.4. Sampled air should pass directly into the sampling train without use of extraneous tubing.

2.3.5. Immediately after sampling, separate the air sampling train into its component tubes, identify each tube as treated or untreated tube and seal each tube with plastic end caps. Also, wrap each sample end to end with official OSHA seals.

2.3.6. With each batch of samples, submit at least one blank *p*-anisidine treated tube from the same lot used for samples. This tube should be subjected to the same treatment and handling as the samples except that no air is drawn through it.

2.3.7. The presence of phthalic or trimellitic anhydride in the sampling area must be reported to the lab.

2.3.8. Transport the samples and paperwork to the lab for analysis.

2.3.9. Sampling tubes are stable for at least 30 days if shielded from light and stored in a freezer.

2.4. Breakthrough (Breakthrough for the purposes of this study will be defined as the presence of the maleic anhydride derivative on the "B" section of the treated sorbent tube).

2.4.1. Vapor generation system

A vapor generation system for maleic anhydride was constructed by filling an empty silanized sorbent tube with glass wool and placing it in front of the treated XAD-2 tube. A chloroform solution of maleic anhydride was injected onto the glass wool and quantitatively leached off by drawing dry air through the system. This gave experimental control over both the quantity of maleic anhydride and the flow rate while eliminating the possible hydrolysis of maleic anhydride before contact is made with the treated tube. (Section 4.5.)

2.4.2. Laboratory experiments consisted of the following: 1) drawing 60 L of humid air through the treated sorbent tube to simulate a humid sampling atmosphere, 2) transferring the tube to the dry air vapor generator and introducing a known quantity of maleic anhydride, and 3) subsequently passing more humid air through the sampling system. It is this volume of humid air which is presented in the third column of Table 4.5.2.

2.4.3. Breakthrough of the maleic anhydride from the "A" section to the "B" section of the tube is primarily a function of the flow rate at which the derivative will all form on the "A" section and secondarily a function of the air volume at which migration of the derivative will take place. At 0.1 L/min, all of the derivative will be formed on the "A" section and up to 150 L of humid air could be pulled without appreciable migration. A 20-L air volume at 0.1 L/min falls well within the parameters of providing a sensitive detection limit, quantitative recovery of maleic anhydride and a practical sampling period of 200 min. This study used an amount of maleic anhydride approximately equal to 2 times the PEL based on the recommended air volume. (Section 4.5.)

2.5. Desorption efficiency

The average desorption efficiency from *p*-anisidine treated XAD-2 tubes with 9.5, 19.1, and 41.5 µg of maleic anhydride injected as the *p*-anisidine derivative was 96.2%. The spiked tubes represent a range from 0.48 mg/m^3 to 2.08 mg/m^3 based on the recommended air volume. (Section 4.6.)

2.6. Recommended air volume and sampling rate

2.6.1. The recommended air volume is 20 L.

2.6.2. The recommended sampling rate is 0.1 L/min.

2.7. Interferences (sampling)

Generic anhydrides present in the sampled atmosphere will react with the *p*-anisidine and thereby reduce the amount available for the derivatization of maleic anhydride. Hence, both phthalic and trimellitic anhydride should be considered as potential sampling interferences.

2.8. Safety precautions (sampling)

2.8.1. Observe due care when working with the sharp ends of the air sampler.

2.8.2. Attach the sampling equipment to the worker in such a manner that it will not interfere with work performance.

2.8.3. Assure that the untreated XAD-2 backup tube is securely in place to collect any *p*-anisidine lost from the treated tube.

2.8.4. Follow all safety practices that apply to the work area being sampled.

3. Analytical Method

3.1. Apparatus

3.1.1. A High Performance Liquid Chromatograph interfaced to a UV absorbance detector.

3.1.2. A reverse phase C_{18} liquid chromatographic column.

3.1.3. An electronic integrator or other suitable method to measure peak magnitude.

3.1.4. An analytical balance.

3.1.5. 2-mL vials with Teflon-lined caps.

3.1.6. Syringes, 50-µL for sample injections.

3.1.7. Pipets of convenient sizes for diluting standards and a 1-mL pipet or dispenser for the desorbing solvent.

3.1.8. Volumetric flasks of convenient sizes for diluting standards.

3.1.9. Shaking device for desorption of samples.

3.2. Reagents

3.2.1. *p*-Anisidine, reagent grade.

3.2.2. Maleic anhydride, reagent grade.

3.2.3. Methyl alcohol, chromatographic grade.

3.2.4. Water, LC grade.

3.2.5. Phosphoric acid.

3.2.6. Chloroform, chromatographic grade.

3.3. Sample preparation

3.3.1. The status of the OSHA seal on each sample is noted and recorded as intact, broken, or none.

3.3.2. The field and laboratory numbers are checked against those on the sample identification sheets.

3.3.3. The "A" and "B" sections of the treated sorbent tube should be transferred to separate 2-mL vials. The front glass wool plug should be included with the "A" section and great care should be exercised with the sorbent beads closest to the front of the tube as this is where the maleic anhydride derivative concentration will be greatest.

3.3.4. To each vial, add 1.0 mL of methanol and seal immediately with Teflon-lined caps.

3.3.5. The vials should be shaken vigorously for 60 min.

3.4. Standard preparation

3.4.1. Neat standard preparation is accomplished by dissolving stoichiometric quantities of *p*-anisidine and maleic anhydride in separate solutions of chloroform and then com bining the two solutions. The derivative will precipitate as a yellow solid. After washing the solid several times with chloroform to remove excess *p*-anisidine and maleic anhydride, evaporate the chloroform using a vacuum or under a nitrogen gas stream and use the solid as neat standard.

3.4.2. Weigh out the derivative into a volumetric such that the concentration of the stock solution is no more than 1 mg/mL after addition of methanol. Sonication, shaking and/or warming this standard may facilitate the derivative's dissolution in the methanol. Dilute to the working range with methanol. A derivative solution of 45.1 µg/mL is equivalent to a maleic anhydride air concentration of 1.0 mg/m^3 for a 20-L air sample desorbed with 1 mL of methanol. This amount is uncorrected for the desorption efficiency.

3.4.3. Standards are stored in dark bottles under refrigeration.

3.5. Analysis

3.5.1. LC conditions - Waters M-6000A HPLC Pump
column: Nucleosil C_{18} (25 cm × 4.6 mm)
eluent: 50% MeOH, 50% H_2O, 0.1% phosphoric acid

flow	1.5 mL/min
pressure:	2600 psi
injection volume:	25 µL
elution time:	6.5 min

3.5.2. Detector conditions - Waters 440 Dual Wavelength Absorbance Detector
wavelength: 313 nm primary, 254 nm secondary
attenuation: vary as per need

3.5.3. Chromatogram (Figure 4.3.)

3.5.4. Peak magnitude is measured by electronic integration or other suitable means.

3.5.5. An external standard procedure is used to prepare a calibration curve from the analysis of at least six different standards diluted from two separate weighings.

3.5.6. Bracket the samples with analytical standards.

3.6. Interferences (analytical)

3.6.1. Any collected compound that has the same LC retention time as the derivative and exhibits a UV absorbance at 313 nm is an interference.

3.6.2. LC parameters may be changed to circumvent most interferences.

3.6.3. Retention time alone is not proof of a chemical identity. Samples should be confirmed by GC/MS or other suitable means when required.

3.7. Calculations

3.7.1. The integrator value in area units for each standard is plotted against its concentration in µg/mL and a calibration curve using the best straight line through the points obtained.

3.7.2. The concentration values in µg/mL for both "A" and "B" sections of the treated XAD-2 tube are determined from the calibration curve, added together and corrected for the desorption efficiency.

3.7.3. The air concentration of maleic anhydride (MA) for a sample is calculated by the following equation:

$$mg/m^3 = (A)(B)(C)/D$$

where A = µg/mL derivative from Section 3.7.2.
B = desorption volume in mL
C = molecular weight ratio maleic anhydride/derivative = 0.443
D = sample air volume in liters

3.8. Safety precautions (analytical)

3.8.1. Maleic anhydride is an extremely dangerous irritant with a high vapor pressure that readily sublimes at room temperature. Avoid all skin contact and use only in a well ventilated area.

3.8.2. *p*-Anisidine is a suspected carcinogen. Avoid all skin contact with both the chemical and the treated sorbent beads. Use in a well ventilated area.

3.8.3. Confine the use of solvents to a fume hood.

3.8.4. Wear safety glasses in all laboratory areas.

4. Backup Data

4.1. Detection limit

A 0.22 mg/mL derivative standard is prepared by injecting 4.0 µL of a 55.10 µg/mL derivative

standard into 1.0 mL of MeOH. Twenty-five microliters of this standard injected 5 times gave the following values. Peak heights were used because integration was poor at this level. A typical chromatogram can be seen in Figure 4.1.

Table 4.1
Detection Limit Data

peak height (cm)	statistics
0.54	
0.54	$\bar{x}$ = 0.586
0.62	SD = 0.49
0.58	CV = 0.083
0.65	

4.2. Reliable quantitation limit

Exactly 4.0 µL of a 55.10 µg/mL standard of the derivative was injected onto five *p*-anisidine treated XAD-2 tubes and allowed to stand capped overnight. Desorption in 1 mL of MeOH, shaking for 1 h and analysis by injecting 25 µL into HPLC apparatus produced the following results.

Table 4.2
Reliable Quantitation Limit Data

peak height (cm)	% recovered (0.586 cm = 100%)	statistics
0.50	85.3	
0.49	83.6	$\bar{x}$ = 93.1
0.58	98.9	SD = 7.93
0.58	98.9	±1.96 SD = ±15.6
0.58	98.9	

4.3. Precision

The data in Table 4.3. represent replicate 25-µL injections of three standard solutions. The concentrations of the standards were 9.53 19.05 and 41.50 µg/mL maleic anhydride as the *p*-anisidine derivative. These values would approximate 0.5, 1, and 2 times the PEL at the recommended air volume. A typical integration by the HP 3354 Laboratory Data System is shown in Figure 4.3.

Table 4.3
Precision of the Analytical Method

× target concentration	0.5×	1×	2×
µg MA	9.53	19.05	41.50
µg adduct formed	21.48	42.96	93.59
area counts	501687	1030920	2045560
	507853	1036560	2062770
	507141	1017060	2064960
	503126	1054290	2072360
$\bar{x}$	504952	1034708	2061413
SD	3011	15413	11337
CV	0.0060	0.0149	0.0055
pooled CD	0.0098		

4.4. Sensitivity

A calibration curve is shown in Figure 4.4. using data from Table 4.3. The slope of the curve indicates the sensitivity of the method over the range of 0.5 to 2 times the PEL based on the recommended air volume.

4.5. Breakthrough

4.5.1. The data in Table 4.5.1. represent the vapor generation unit's efficiency at producing maleic anhydride vapors at quantitative levels. Ten microliters of a 1905 µg/mL maleic anhydride standard was injected onto the glass wool tube and then dry air was pulled through the system.

Table 4.5.1.
Vapor Generation System Efficency

tube no.	air volume (L) dry air at 0.2 L/min	% recovered as derivative from treated tube
1	2	98
2	3	100
3	5	96
4	4	100
5	4	96
6	4	100

4.5.2. The data in Table 4.5.2. represent areas obtained from "A" and "B" sections of treated tubes using different flow rates for the vapor generation. The percent B figure is indicative of breakthrough being flow related.

Table 4.5.2.
Variation of Sampler Flow Rates During Vapor Generation

number	vapor generation 3-L vol. dry air flow rate (L/min)	humid air volume (L)	% found on "B" section
1	0.2	60	2.6
2	0.2	60	2.3
3	0.1	60	0.08
4	0.1	60	0.06
5	0.1	150	0.15

4.6. Desorption

Samples representing maleic anhydride concentrations of 0.49, 0.98 and 1.95 mg/m^3, based on a 20-L air volume and a 1-mL desorption volume, were prepared by injecting 4, 8, and 16 µL of a MeOH solution of derivative, whose concentration was 5510 µg/mL, onto *p*-anisidine treated tubes. The samples were stored for 24 h and analyzed.

Table 4.6
Desorption Efficiency Data

mg/m^3	0.49	0.98	1.95
desorption efficiency %	89.78	100.00	99.62
	92.63	100.13	100.10
	94.57	99.46	99.10
	92.94	97.83	97.48
	87.61	98.02	92.71
$\bar{x}$	91.51	99.09	97.80
overall $\bar{x}$	96.01		

4.7. Storage

Storage samples were prepared by injecting 8 µL of a 55.10 mg/mL standard of the derivative onto 36 treated XAD-2 tubes. This value approximates the PEL level for maleic anhydride based on the recommended air volume. The data in Table 4.7. represent the recovery of derivative from treated XAD-2 tubes stored at the ambient temperature (approximately 23°C) and at reduced temperature (approximately 5°C). A graphical representation can be found in Figures 4.7.1. and 4.7.2.

Table 4.7.
Storage Tests

storage time (days)	% recovery (refrigerated)			(ambient)		
0	94.0	94.0	98.0	99.0	95.0	96.0
3	94.0	93.0	96.0	95.0	92.0	86.0
7	78.0	83.0	95.0	100.0	88.0	94.0
10	99.0	99.0	99.0	99.0	93.0	91.0
14	92.0	99.0	93.0	98.0	87.0	94.0
15	92.0	98.0	94.0	101.0	102.0	89.0

4.8. The data in Table 4.8. represent the esterification of maleic acid in isopropanol under various conditions. A 0.5-mL aliquot of a 222 ng/µL solution of maleic acid in isopropanol was added to each of 8 vials containing 4.5 mL of isopropanol with the pH adjusted as follows:

1) 3 vials adjusted to pH 1.8 with H_2SO_4
2) 3 vials left neutral
3) 2 vials adjusted to pH 13.0 with KOH

All vials were subjected to a temperature of 41°C over a 36-h period and analyzed for the monoisopropyl ester of maleic acid by HPLC. Results are shown in Table 4.8. Note that the ester was previously believed to have been formed only from maleic anhydride in isopropanol. A significant doubt could be raised as to the validity of maleic anhydride air concentration results determined in this manner.

Table 4.8
Esterification of Maleic Acid in IPA Under Various Conditions

vial no.	pH condition	% maleic acid esterified (vs. 22.2 ng/µL std)
1	acid	12.2
2	acid	12.4
3	acid	13.3
4	neutral	2.7
5	neutral	3.0
6	neutral	3.3
7	basic	1.6
8	basic	1.6

4.9. Preparation of *p*-anisidine coated XAD-2 tubes

4.9.1. Reagents

Methylene chloride, chromatographic grade.

p-Anisidine. Prepare a solution containing 2 mg/mL *p*-anisidine in methylene chloride. Store in a dark bottle and refrigerate.

XAD-2 sorbent tubes; 7-cm length, 6-mm o.d., 80-mg front, 40-mg back. SKC catalog no. 226-30.

4.9.2. Preparation

The tubes are prepared by breaking both ends of untreated XAD-2 tubes open and adding 250 µL of the *p*-anisidine/methylene chloride solution to the tube. Both sections of the tube should be uniformly wetted by the solution. The methylene chloride is evaporated under a reduced atmosphere, the tubes are capped and stored in a freezer.

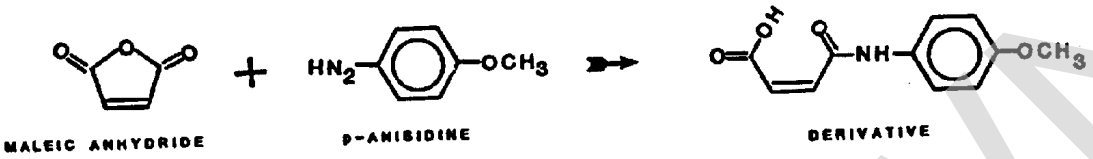

Figure 1.1.1. Derivatization reaction between maleic anhydride and *p*-anisidine.

Figure 1.1.4. Structure of maleic anhydride and of the *p*-anisidine derivative of maleic anhydride.

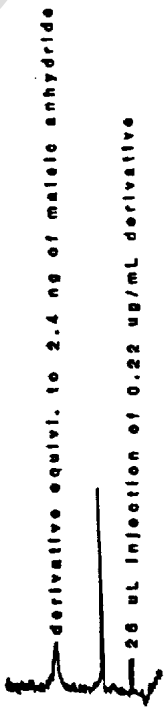

Figure 4.1. Detection limit of the analytical procedure.

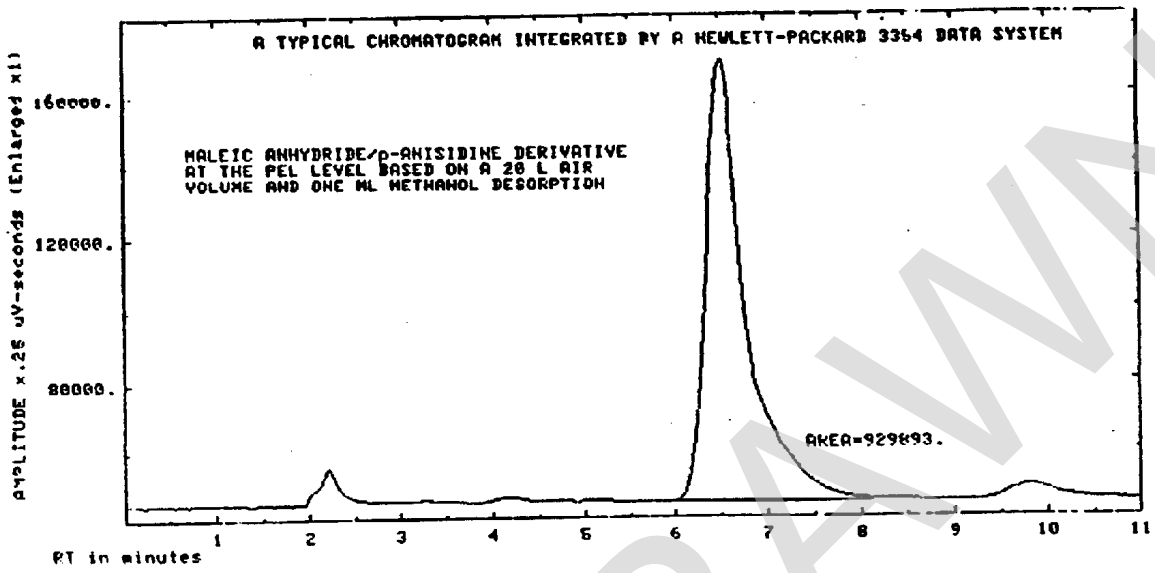

Figure 4.3. A typical chromatogram for the *p*-anisidine derivative of maleic anhydride.

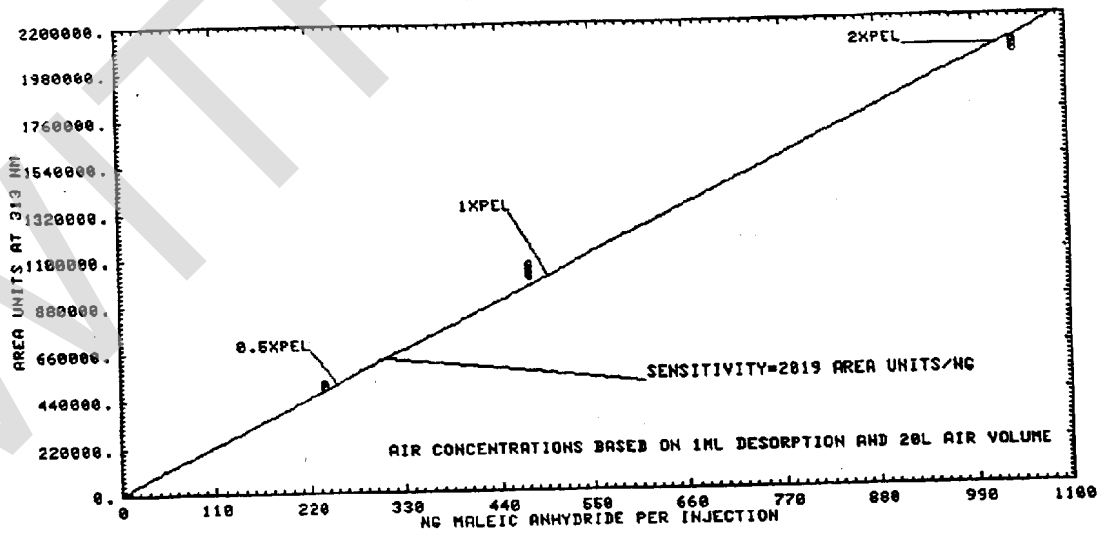

Figure 4.4. Calibration curve.

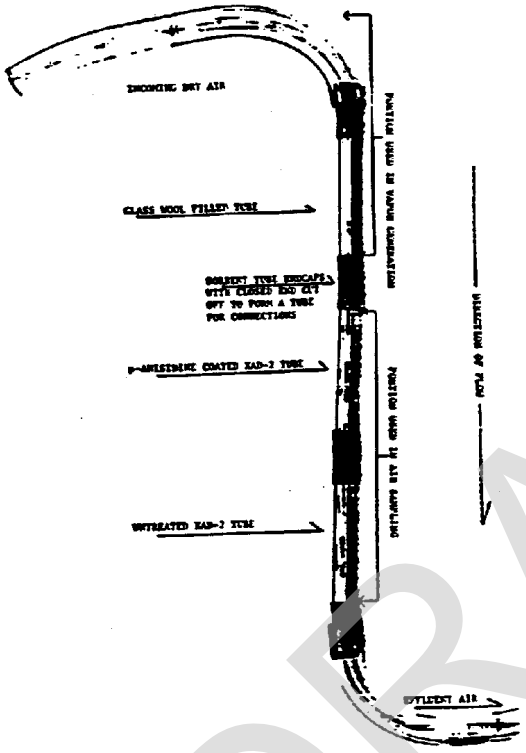

Figure 4.5. Vapor generation and sampling system for maleic anhydride.

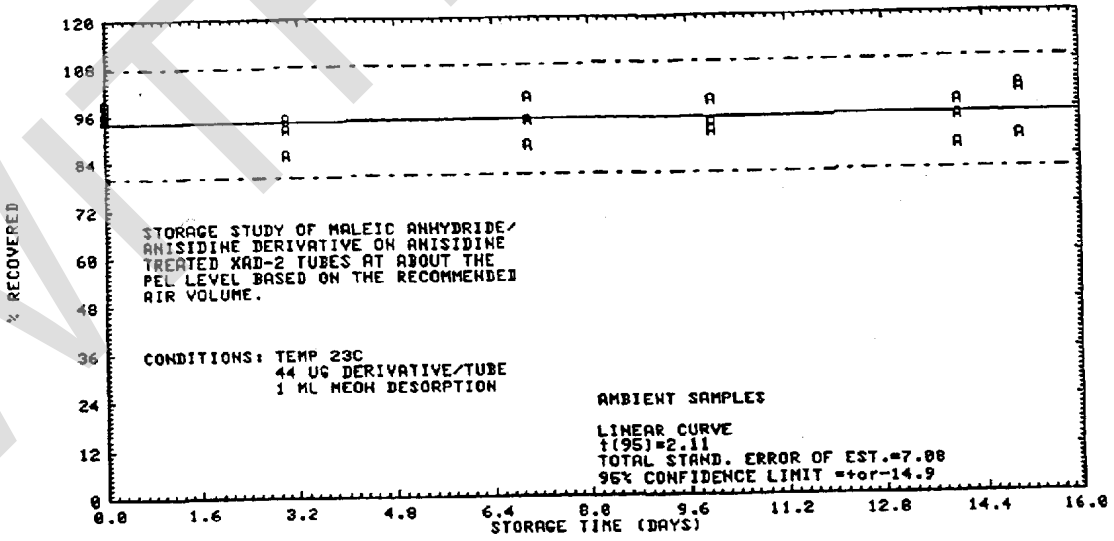

Figure 4.7.1 Ambient temperature storage test.

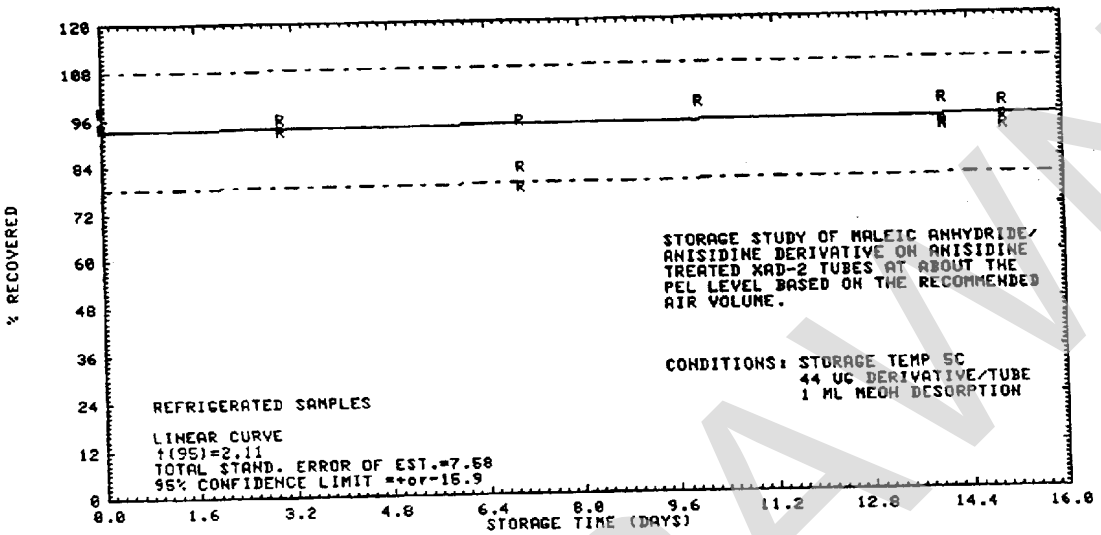

Figure 4.7.2. Refrigerated temperature storage test.

5. References

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