

VANADIUM OXIDES

7504

(1): V ₂ O ₅	MW: (1): 181.88	CAS: (1) 1314-62-1	RTECS: (1) YW2450000 (dust)
(2): V ₂ O ₃	(2): 149.88	(2) 1314-34-7	(1) YW2460000 (fume)
(3): NH ₄ VO ₃	(3): 116.99	(3) 7803-55-6	(2) YW3050000
			(3) YW0875000

METHOD: 7504, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 August 1987
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<p>OSHA: C 0.5 mg/m³ (as V) (V₂O₅ Dust); C 0.1 mg/m³ (as V) (V₂O₅ Fume); 1 mg/m³ (FeV)</p> <p>NIOSH: C 0.05 mg/m³/15 min (as V₂O₅)</p> <p>ACGIH: 0.05 mg/m³ (respirable, as V₂O₅)</p>	<p>PROPERTIES: solids; MP 658 °C (1); MP 1967 °C (2)</p>
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SYNONYMS: (1): vanadic anhydride; vanadium pentoxide;
(2): vanadic oxide; vanadium sesquioxide; vanadium trioxide.

SAMPLING	MEASUREMENT
<p>SAMPLER: CYCLONE + FILTER (10-mm cyclone or Higgins-Dewell (HD) + 5-µm PVC membrane)</p> <p>FLOW RATE: HD cyclone: 2.2 L/min nylon cyclone: 1.7 L/min</p> <p>VOL-MIN: 200 L @ 0.5 mg/m³</p> <p>-MAX: 1000 L</p> <p>SHIPMENT: routine</p> <p>SAMPLE STABILITY: stable</p> <p>BLANKS: 2 to 10 field blanks per set</p> <p>BULK SAMPLE: high-volume respirable (preferred) or settled dust to identify interferences</p>	<p>METHOD: X-RAY POWDER DIFFRACTION</p> <p>ANALYTE: vanadium pentoxide, vanadium trioxide, or ammonium metavanadate</p> <p>SAMPLE PREPARATION: dissolve filter in tetrahydrofuran; redeposit on Ag filter</p> <p>XRD: Cu target X-ray tube optimize for intensity; 1° 2θ slit graphite monochromator scintillation detector integrated intensity with background subtraction slow step scan (10 seconds/0.02° 2θ)</p> <p>CALIBRATION: standard suspensions of analytes deposited on Ag filter</p> <p>RANGE: 0.1 to 2 mg V per sample</p> <p>ESTIMATED LOD: Table 1</p> <p>PRECISION (\hat{S}_r): Table 1</p>
ACCURACY	
<p>RANGE STUDIED: (1): 0.8-2.6 mg/m³; (2): 0.2-2.6 mg/m³ [1]</p> <p>BIAS: Table 1</p> <p>OVERALL PRECISION (\hat{S}_{rT}): Table 1</p> <p>ACCURACY: ± 26 to 62%</p>	

APPLICABILITY: The working range is 0.2 to 4 mg/m³ (as V) for a 500-L air sample. The method will determine V₂O₅, V₂O₃, and NH₄VO₃ separately on the same sample.

INTERFERENCES: α-Quartz (likely to be found in mineral samples) interferes (Table 1) but alternative diffraction lines are available. Most other compounds likely to be in the sample do not interfere [1].

OTHER METHODS: This replaces P&CAM 364 [2]. Methods S391 (Vanadium by graphite atomizer AAS) and 7300 (Elements by ICP-AES) determine total V.

REAGENTS:

1. V_2O_5 , V_2O_3 or NH_4VO_3 ($\geq 99\%$). Grind in freezer mill. Sieve through 10- μ m sieve in isopropanol (for V_2O_3 or NH_4VO_3) or acetonitrile (for V_2O_5). Dry at 110 °C for one hour. Store in desiccator.
2. Tetrahydrofuran (THF), reagent grade.*
3. Isopropanol, reagent grade.
4. Acetonitrile, reagent grade.
5. Glue or tape for securing silver filters to XRD holders.
6. Desiccant.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler:
 - a. Filter: 37-mm diameter, 5.0- μ m pore size, polyvinyl chloride filter supported with backup pad in a two-piece, 37-mm cassette filter holder (preferably, conductive) held together by tape or cellulose shrink band.
NOTE: Test PVC filter for THF digestion (see Step 4).
 - b. Cyclone: 10-mm nylon or Higgins-Dewell (HD), or equivalent.
 - c. Sampling head holder: Holder must keep the cassette, cyclone, and coupler together rigidly so that air enters only at the cyclone inlet.
2. Bulk sampler: PVC membrane filter, 37-mm, 5- μ m pore size in two-piece filter cassette. Sample closed face at 3 L/min.
3. Sampling pumps: HD cyclone, 2.2 L/min; nylon cyclone, 1.7 L/min; and bulk sampler, 3 L/min.
4. Filters, silver membrane, 25-mm diameter, 0.45- μ m pore size (Osmonics, Inc., Nuclepore, Poretics, or equivalent).
5. X-ray powder diffractometer with copper target X-ray tube, graphite monochromator, and scintillation detector.
6. Reference specimen (mica, Arkansas stone or other stable standard. Source: Gem Dugout, State College, PA, 16801) for data normalization.
7. Filtration apparatus and side arm vacuum flask, with 25-mm filter holders.
8. Sieve, 10- μ m, for wet-sieving.
9. Centrifuge tubes, wide-mouth, 40-mL.
10. Ultrasonic bath or probe.
11. Analytical balance (0.01 mg).
12. Magnetic stirrer with thermally-insulated top.
13. Bottles, reagent, 1-L, with ground glass stoppers.
14. Drying oven.
15. Polyethylene wash bottle.
16. Desiccator.
17. Pipets, TD, 2- to 25-mL.

SPECIAL PRECAUTIONS: THF is extremely flammable and should be used in a fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at $1.7 \pm 5\%$ L/min with a nylon cyclone or $2.2 \pm 5\%$ L/min with an HD cyclone for a total sample size of 200 to 1000 L. Do not exceed 2 mg dust loading on the filter.
NOTE: Do not allow the sampler assembly to be inverted at any time. Turning the cyclone to more than 90° from vertical may deposit over-sized material from the cyclone body onto the filter.
3. Obtain an area high-volume bulk sample in the vicinity of the personal sampling area.

SAMPLE PREPARATION:

4. Place sample filter in a centrifuge tube. Add 10 mL THF. Place centrifuge tube in ultrasonic bath for 10 min.
NOTE: The filter should dissolve almost instantaneously.
5. Mount a silver filter in the filtration apparatus. Attach the funnel securely over the entire filter circumference. With no vacuum, pour 2 to 3 mL THF onto the filter. Pour the sample suspension from the centrifuge tube into the funnel. Rinse centrifuge tube twice with 5-mL portions of THF, adding rinses to the funnel, and apply vacuum.
6. Control filtration rate to keep liquid level near top of funnel during filtering. Do not wash the walls or add THF to the funnel when the liquid level is lower than 4 cm above the filter. Leave vacuum on after filtration for sufficient time to dry the filter. Transfer filter with forceps to sample holder for XRD analysis.

CALIBRATION AND QUALITY CONTROL:

7. Select six silver membrane filters as media blanks, randomly from the same box of filters to be used for depositing the samples. Mount each media blank on the filtration apparatus and apply vacuum to draw 5 to 10 mL THF through the filter. Remove, let dry and mount on XRD holders.
NOTE: These will be used to test for sample self-absorption.
8. Prepare standard suspensions.
 - a. Weigh 10- and 50-mg portions of the dry analytes to the nearest 0.01 mg. Quantitatively transfer to 1-L glass-stoppered bottles. Add 1.00 L acetonitrile (for V_2O_5) or isopropanol (for V_2O_3 or NH_4VO_3).
 - b. Disperse the powder in the liquid using an ultrasonic probe or bath for 20 min. Immediately move the flask to a magnetic stirrer and add a stirring bar to the suspension. Allow the solution to return to room temperature before withdrawing aliquots.
9. Prepare a series of standard filters over the range 0.05 to 2 mg V per sample.
 - a. Mount a filter on the filtration apparatus. Wet the filter with ca. 3 mL of acetonitrile (for V_2O_5) or isopropanol (for V_2O_3 or NH_4VO_3).
 - b. Turn off the stirrer and shake the bottle vigorously by hand. Immediately remove the lid and withdraw an aliquot (2 to 25 mL) from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension. If more than the desired aliquot is withdrawn, return all of the suspension to the bottle, rinse and dry the pipet, and take a new aliquot.
 - c. Transfer the aliquot from the pipet to the funnel. Keep the tip of the pipet near the surface but not submerged in the suspension. Rinse the pipet with ca. 5 mL of acetonitrile (for V_2O_5) or isopropanol (for V_2O_3 or NH_4VO_3), draining the rinse into the funnel. Repeat the rinse three more times.
 - d. Apply vacuum and rapidly filter the suspension. Leave vacuum on until filter is dry. Do not wash down sides of funnel after deposit is in place (to avoid rearranging the material on the filter).
 - e. Transfer the filter to the XRD sample holder.

10. Perform step scans on the standards and reference specimen using the same conditions as for samples. Use steps 12 and 13 to determine normalized intensity, \hat{I}_x^o , for each peak measured. Use exactly the same normalization factor, N, as for samples (step 13).
11. Prepare calibration graph (\hat{I}_x^o vs. mg of each standard). Determine slope, m (counts/ μ g).

NOTE 1: The intercept with the \hat{I}_x^o axis should be ca. zero. A large negative intercept indicates an error in determining background (e.g., incorrectly measuring the baseline, or interference by another phase at the angle of background measurement). A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

NOTE 2: Poor repeatability at a given level indicates problems in the sample preparation technique and new standards should be made. Eliminate curvature with absorption corrections based on the mass absorption coefficient of the analyte (step 15, or from Tables 2 through 4).

MEASUREMENT:

12. Obtain a qualitative X-ray diffraction scan (broad 2θ range) of high-volume sample to determine the presence of interferences.

NOTE: If quantitative analysis is to be done on the bulk sample, wet-sieve it first through a 10- μ m sieve.
13. Mount the filter (sample, standard, or blank) in the XRD instrument and perform the following:
 - a. Determine net intensity, I_x^o , of the reference specimen before filter is scanned. Select a convenient scale factor, N, which is approximately equivalent to the net count for the reference specimen peak; use this for all analyses.
 - b. Step-scan the most intense, interference-free diffraction peak of each compound to be determined, integrating the counts.

NOTE: Useful analytical lines for the analytes are given in Table 1. Use strongest line of the analyte which does not have a matrix interference. Avoid lines in the proximity of Ag (JCPDS #4-0783 [3]) and AgCl (JCPDS #31-1238 [4]) (the latter often exists on the surface of silver filters).
 - c. Measure the background on each side of the peak for one-half the time used for peak scanning. Add the counts from each side to obtain total (average) background.
 - d. Calculate net intensity, I_x , (different between peak integrated count and total background count).
 - e. Calculate and record the normalized intensity for the analyte peak on each sample and standard:

$$\hat{I}_x = \left(\frac{I_x}{I_r^o} \right) \times N.$$

For each media blank, determine the net count for the analyte diffraction peak. Calculate the average normalized intensity, \hat{I}_b , for the 6 media blanks.

- f. Determine net count, I_{Ag} , of an interference-free silver peak on the filter following the same procedure. Scan times should be shorter for the silver peak (e.g., about 5% of scan times for analyte peaks) and should be consistent throughout the method. For each media blank, determine the net count for the silver peak. Calculate the average value, I_{Ag}^o , for the 6 media blanks.

NOTE: Normalizing to the reference specimen intensity compensates for long-term drift in X-ray tube intensity. If intensity measurements are stable, the reference specimen may be run less frequently. In this case, the net intensities of the analyte, blank, and silver peaks (I_x , I_b , and I_{Ag}) should be normalized to the most recently measured reference intensity.

14. Scan each field blank over the same 2θ range used for the analyte and silver peaks. The analyte peak should be absent. The normalized intensity of the silver peak of the field blanks should match that of the media blanks.

NOTE: These analyses serve only to verify that contamination of the filters has not occurred.

CALCULATIONS:

15. Calculate absorption correction factors (Tables 2, 3, and 4) [5]:

$$f(T) = \frac{-R \ln T}{1 - T^R}$$

where: $R = (\sin \theta_{Ag})/(\sin \theta_x)$ and

$T = \hat{I}_{Ag}/\hat{I}_{Ag}^0$ = transmittance of sample.

16. Calculate concentration, C, of analyte in air volume sampled, V (L):

$$C = \frac{([\hat{I}_x \cdot f(T)] - \hat{I}_b)}{m \cdot V}, \text{ mg/m}^3.$$

where: \hat{I}_x = normalized intensity of sample peak
 \hat{I}_b = normalized intensity of blank
 m = slope of calibration graph, counts/ μg

EVALUATION OF METHOD:

VANADIUM PENTOXIDE: The V_2O_5 method was evaluated [1] with both spiked and generated samples of V_2O_5 . With the spiked samples, using the 31.05° line and loadings of 433 to 699 $\mu\text{g}/\text{filter}$, the overall \hat{S}_{rT} was 8.3% with an average bias of -7.3%. Five sets of generated samples, ranging from 184 to 2400 $\mu\text{g V}_2\text{O}_5$ per filter, gave an average bias of 10.12% with a pooled \hat{S}_r of 6.9%. The generated samples used an aerosol which had been sized with a cyclone with characteristics similar to the 10-mm nylon cyclone; however, individual 10-mm nylon cyclones on the cassettes were not used.

VANADIUM TRIOXIDE: The method was evaluated [1] with both spiked and generated samples of V_2O_3 . With the spiked samples, and the 24.39° line and loadings of 107 to 321 μg per filter, the overall \hat{S}_r was 8.2% with an average bias of 12.2%. Five sets of generated samples, from atmospheres of 0.24 to 2.57 $\text{mg V}_2\text{O}_3/\text{m}^3$, gave an average bias of -10.1% with a pooled \hat{S}_r of 13.4% (24.39° line). As above, the samples were generated by using an aerosol, which had been sized by means of a cyclone with characteristics similar to the 10-mm nylon cyclone; however, individual 10-mm nylon cyclones on the cassettes were not used.

AMMONIUM METAVANADATE: The method was evaluated [1] with spiked NH_4VO_3 samples over the range of 109 to 277 $\mu\text{g NH}_4\text{VO}_3$ per filter, and with samples generated as described above, with atmospheres of 0.037 to 0.435 mg/m^3 of NH_4VO_3 . The $18.10^\circ 2\theta$ line provided an overall \hat{S}_{rT} of 26.3%, with a bias of -10.2% over the generation range. The stability of the compound is critical and analysis should occur within two weeks of sampling, unless refrigeration storage is employed. Sampling is quantitative using the two-filter technique described.

For the analytes, biases of spiked samples were determined by assuming the volume of suspension as "true." Biases of generated samples were determined by assuming that the concentration obtained from the ICP-AES analysis of monitor AAWP filters was the "true" concentration.

Instrumental imprecision increases with smaller depositions. For low loading levels, higher counting times will increase precision, as should sample spinning. The stated lower limit of quantitation of ca. 100 µg per analyte when the recommended analytical lines were used is an estimate based on numerous spiked and generated sample experiments.

REFERENCES:

- [1] T. Carsey, "Quantitation of Vanadium Oxides in Airborne Dusts by X-Ray Diffraction," Anal. Chem., **57**: 2125-2130 (1985).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 8 (unpublished, 1982).
- [3] Powder Diffraction File Search Manual, International Centre for Diffraction Data (JCPDS), Swarthmore, PA, 19081, p. 809 (1981).
- [4] *Ibid.*, p. 810.
- [5] M. Abell, D. Dollberg, J. Crable, "Quantitative Analysis of Dust Samples from Occupational Environments Using Computer-Automated X-ray Diffraction," in Advances in X-Ray Analysis, Vol. 24, Plenum, p. 37 (1981).
- [6] E. Bertin: Principles and Practice of X-ray Spectrometric Analysis, 2nd Ed., Plenum, New York, p. 471 (1975).
- [7] Criteria for a Recommended Standard...Occupational Exposure to Vanadium, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-222 (1977).

METHOD REVISED BY:

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Table 1. Characteristics of analytical lines.

d-space (A)	Relative Intensity ¹	Peak 2 θ ¹	Scan		Detection Limit ² (μg)	Cali- bration Slope (ct/ μg)	Preci- sion ³ (% \hat{S}_{rT})	Bias ⁴ (%)	Comments
		Min 2 θ	Max 2 θ						
<u>Vanadium Pentoxide (PDF # 9-0387)</u>									
4.38	100	20.27	19.4	20.9	4	205.0	11.5	-28.9	α -Quartz interf. 20.85 $^{\circ}2\theta$
4.09	35	21.73	20.9	22.4	13	57.1	-	-	α -Quartz interf. 20.85 $^{\circ}2\theta$
3.40	90	26.21	25.5	26.6	10	54.2	11.3	-22.3	α -Quartz interf. 26.69 $^{\circ}2\theta$
2.88	65	31.05	30.3	31.4	9	73.0	8.3	10.1	good
2.185	17	41.32	40.5	41.6	28	24.9	-	-	weak; V_2O_3 interf. 41.42 $^{\circ}2\theta$
<u>Vanadium Trioxide (PDF # 26-278)</u>									
3.65	60	24.39	24.75	23.5	6	75.1	14.7	-10.1	good
2.70	80	33.18	33.5	32.5	5	78.3	16.0	0.0	AgCl interf. 32.32 $^{\circ}2\theta$
2.47	60	36.37	36.8	35.7	9	70.6	15.6	14.2	α -Quartz interf. 36.53 $^{\circ}2\theta$
2.18	20	41.42	41.7	40.6	62	11.7	-	-	weak; V_2O_5 interf. 41.32 $^{\circ}2\theta$
1.83	25	49.83	50.4	49.3	21	30.2	-	-	weak
1.69	100	54.28	54.5	53.3	5	91.2	20.7	-10.1	good
1.43	30	65.25 ⁵							
<u>Ammonium Metavanadate (PDF # 9-411)</u>									
5.88	50	15.07	14.3	15.4	7	133.8	24.0	11.4	good
4.90	75	18.10	17.7	18.2	7	101.4	26.3	-10.2	good
4.14	95	21.46	21.0	21.8	10	49.8	-	13.2	weak; V_2O_5 interf. 21.73 $^{\circ}2\theta$
3.77	40	23.60	23.1	23.9	18	38.5	-	-	weak; V_2O_5 interf. 41.32 $^{\circ}2\theta$
3.164	100	28.21 ⁶			50.3	-	-	-	AgCl interf. at 27.88 $^{\circ}2\theta$
2.912	60	30.74	30.1	30.9	21	33.9	-	-	weak
2.628	45	34.12	33.5	34.4	20	36.0	-	-	weak

¹ From Reference [7], p. 979 ($g = 1.5418\text{\AA}$) $^{\circ}$.² Reference [6].³ $\hat{S}_{rT} = (\text{GRSD}^2 + 0.1667 \cdot \text{SRSD}^2 + .05^2)^{1/2}$, where GRSD is the generated-sample pooled relative standard deviation, and SRSD is the spiked-sample pooled relative standard deviation. This is the estimated relative standard deviation for the total (sampling plus measurement) air-monitoring method.⁴ From generated samples.⁵ Not evaluated because of an Ag interference at 64.48 $^{\circ}2\theta$.⁶ Not evaluated because of an AgCl interference at 27.88 $^{\circ}2\theta$.

Table 2. Matrix absorption correction factors for vanadium pentoxide/silver peaks by degrees two-theta.

		20.27°	26.13°	31.00°			20.27°	26.13°	31.00°
		38.12°	38.12°	38.12°			38.12°	38.12°	38.12°
V ₂ O ₅									
Silver									
T	(T)	f(T)	f(T)	T	f(T)	f(T)	f(T)	f(T)	f(T)
1.00	1.0000	1.0000	1.0000	0.74	1.3053	1.2332	1.1952		
0.99	1.0094	1.0073	1.0062	0.73	1.3203	1.2445	1.2046		
0.98	1.0189	1.0147	1.0124	0.72	1.3356	1.2560	1.2141		
0.97	1.0285	1.0222	1.0187	0.71	1.3512	1.2677	1.2238		
0.96	1.0384	1.0298	1.0251	0.70	1.3672	1.2796	1.2337		
0.95	1.0483	1.0375	1.0317	0.69	1.3835	1.2918	1.2438		
0.94	1.0585	1.0454	1.0383	0.68	1.4002	1.3043	1.2541		
0.93	1.0688	1.0533	1.0450	0.67	1.4172	1.3170	1.2646		
0.92	1.0794	1.0614	1.0518	0.66	1.4346	1.3300	1.2753		
0.91	1.0901	1.0697	1.0587	0.65	1.4524	1.3432	1.2862		
0.90	1.1009	1.0780	1.0658	0.64	1.4706	1.3567	1.2973		
0.89	1.1120	1.0865	1.0729	0.63	1.4892	1.3706	1.3087		
0.88	1.1233	1.0952	1.0801	0.62	1.5083	1.3847	1.3203		
0.87	1.1348	1.1040	1.0875	0.61	1.5278	1.3992	1.3322		
0.86	1.1465	1.1129	1.0950	0.60	1.5478	1.4139	1.3444		
0.85	1.1584	1.1220	1.1026	0.59	1.5682	1.4291	1.3568		
0.84	1.1705	1.1312	1.1103	0.58	1.5892	1.4445	1.3695		
0.83	1.1828	1.1406	1.1182	0.57	1.6107	1.4604	1.3825		
0.82	1.1954	1.1502	1.1261	0.56	1.6327	1.4766	1.3957		
0.81	1.2082	1.1599	1.1343	0.55	1.6553	1.4932	1.4094		
0.80	1.2213	1.1698	1.1425	0.54	1.6784	1.5102	1.4233		
0.79	1.2346	1.1799	1.1509	0.53	1.7022	1.5277	1.4376		
0.78	1.2482	1.1902	1.1595	0.52	1.7266	1.5456	1.4522		
0.77	1.2620	1.2006	1.1682	0.51	1.7516	1.5640	1.4672		
0.76	1.2762	1.2113	1.1770	0.50	1.7774	1.5828	1.4826		
0.75	1.2906	1.2221	1.1860	0.49	1.8039	1.6022	1.4984		

T = Sample transmittance (see step 15)
 f(T) = Sample correction factor (see step 15)

Table 3. Matrix absorption correction factors for vanadium trioxide/silver peaks by degrees two-theta.

V ₂ O ₃	24.39°	33.18°	36.37°	54.28	24.39°	33.18°	36.37°	54.28°		
Silver	38.15°	38.15°	38.15°	38.15°	38.15°	38.15°	38.15°	38.15°		
	T	f(T)	f(T)	f(T)	f(T)	T	f(T)	f(T)	f(T)	f(T)
	1.00	1.00000	1.00000	1.00000	1.00000	0.74	1.25097	1.18219	1.16591	1.11173
	0.99	1.00779	1.00576	1.00527	1.00360	0.73	1.26315	1.19089	1.17379	1.11696
	0.98	1.01571	1.01161	1.01061	1.00725	0.72	1.27558	1.19975	1.18182	1.12228
	0.97	1.02375	1.01753	1.01603	1.01095	0.73	1.28825	1.20877	1.19000	1.12769
	0.96	1.03191	1.02354	1.02152	1.01469	0.70	1.30119	1.21797	1.19832	1.13320
	0.95	1.04021	1.02964	1.02709	1.01849	0.69	1.31439	1.22734	1.20681	1.13880
	0.94	1.04863	1.03583	1.03274	1.02233	0.68	1.32786	1.23689	1.21546	1.14450
	0.93	1.05719	1.04211	1.03847	1.02622	0.67	1.34162	1.24663	1.22427	1.15030
	0.92	1.06589	1.04848	1.04429	1.03016	0.66	1.35567	1.25657	1.23326	1.15621
	0.91	1.07474	1.05494	1.05019	1.03416	0.65	1.37002	1.26670	1.24242	1.16223
	0.90	1.08372	1.06151	1.05617	1.03822	0.64	1.38469	1.27705	1.25177	1.16837
	0.89	1.09286	1.06817	1.06225	1.04232	0.63	1.39968	1.28761	1.26131	1.17462
	0.88	1.10215	1.07494	1.06842	1.04649	0.62	1.41500	1.29839	1.27105	1.18099
	0.87	1.11160	1.08181	1.07468	1.05071	0.61	1.43068	1.30941	1.28100	1.18749
	0.86	1.12122	1.08879	1.08104	1.05500	0.60	1.44672	1.32066	1.29116	1.19411
	0.85	1.13099	1.09589	1.08749	1.05934	0.59	1.46313	1.33216	1.30153	1.20088
	0.84	1.14094	1.10309	1.09405	1.06375	0.58	1.47993	1.34392	1.31214	1.20778
	0.83	1.15107	1.11042	1.10072	1.06823	0.57	1.49713	1.35595	1.32298	1.21483
	0.82	1.16137	1.11786	1.10749	1.07277	0.56	1.51475	1.36825	1.33408	1.22203
	0.81	1.17186	1.12543	1.11437	1.07738	0.55	1.53281	1.38084	1.34542	1.22939
	0.80	1.18255	1.13313	1.12137	1.08206	0.54	1.55133	1.39374	1.35704	1.23691
	0.79	1.19342	1.14096	1.12848	1.08681	0.53	1.57031	1.40695	1.36893	1.24460
	0.78	1.20450	1.14892	1.13571	1.09164	0.52	1.58979	1.42048	1.38111	1.25246
	0.77	1.21579	1.15702	1.14306	1.09654	0.51	1.60978	1.43435	1.39359	1.26051
	0.76	1.22729	1.16526	1.15055	1.10152	0.50	1.63030	1.44858	1.40639	1.26875
	0.75	1.23902	1.17365	1.15816	1.10659	0.49	1.65139	1.46318	1.41952	1.27719

T = Sample transmittance (see step 15)
 f(T) = Sample correction factor (see step 15)

Table 4. Matrix absorption correction factors for ammonium metavanadate/silver peaks by degrees two-theta.

NH ₄ VO ₃		15.07°	18.10°	21.46°	23.60			15.07°	18.10°	21.46°	23.60°
Silver		38.15°	38.15°	38.15°	38.15°			38.15°	38.15°	38.15°	38.15°
T	f(T)	f(T)	f(T)	f(T)	T	f(T)	f(T)	f(T)	f(T)	f(T)	
1.00	1.0000	1.0000	1.0000	1.0000	0.74	1.0000	1.0000	1.0000	1.0000	1.0000	
0.99	1.0126	1.0105	1.0088	1.0081	0.73	1.0126	1.0105	1.0088	1.0081	1.0081	
0.98	1.0254	1.0211	1.0178	1.0162	0.72	1.0254	1.0211	1.0178	1.0162	1.0162	
0.97	1.0384	1.032	1.0270	1.0245	0.71	1.0384	1.0320	1.0270	1.0245	1.0245	
0.96	1.0517	1.043	1.0362	1.0330	0.70	1.0517	1.0430	1.0362	1.0330	1.0330	
0.95	1.0653	1.0542	1.0457	1.0415	0.69	1.0653	1.0542	1.0457	1.0415	1.0415	
0.94	1.0791	1.0656	1.0553	1.0503	0.68	1.0791	1.0656	1.0553	1.0503	1.0503	
0.93	1.0932	1.0773	1.0650	1.0591	0.67	1.0932	1.0773	1.0650	1.0591	1.0591	
0.92	1.1075	1.0891	1.0749	1.0681	0.66	1.1075	1.0891	1.0749	1.0681	1.0681	
0.91	1.1221	1.1011	1.0850	1.0773	0.65	1.1221	1.1011	1.0850	1.0773	1.0773	
0.90	1.1371	1.1134	1.0953	1.0866	0.64	1.1371	1.1134	1.0953	1.0866	1.0866	
0.89	1.1523	1.1259	1.1057	1.0960	0.63	1.1523	1.1259	1.1057	1.0960	1.0960	
0.88	1.1678	1.1386	1.1164	1.1056	0.62	1.1678	1.1386	1.1164	1.1056	1.1056	
0.87	1.1836	1.1516	1.1272	1.1154	0.61	1.1836	1.1516	1.1272	1.1154	1.1154	
0.86	1.1997	1.1648	1.1382	1.1254	0.60	1.1997	1.1648	1.1382	1.1254	1.1254	
0.85	1.2162	1.1783	1.1494	1.1355	0.59	1.2162	1.1783	1.1494	1.1355	1.1355	
0.84	1.2330	1.1920	1.1608	1.1458	0.58	1.2330	1.1920	1.1608	1.1458	1.1458	
0.83	1.2501	1.2060	1.1724	1.1563	0.57	1.2501	1.2060	1.1724	1.1563	1.1563	
0.82	1.2676	1.2202	1.1842	1.1669	0.56	1.2676	1.2202	1.1842	1.1669	1.1669	
0.81	1.2855	1.2348	1.1963	1.1778	0.55	1.2855	1.2348	1.1963	1.1778	1.1778	
0.80	1.3038	1.2496	1.2086	1.1889	0.54	1.3038	1.2496	1.2086	1.1889	1.1889	
0.79	1.3224	1.2647	1.2211	1.2002	0.53	1.3224	1.2647	1.2211	1.2002	1.2002	
0.78	1.3414	1.2801	1.2338	1.2116	0.52	1.3414	1.2801	1.2338	1.2116	1.2116	
0.77	1.3609	1.2959	1.2468	1.2233	0.51	1.3609	1.2959	1.2468	1.2233	1.2233	
0.76	1.3807	1.3120	1.2601	1.2353	0.50	1.3807	1.3120	1.2601	1.2353	1.2353	
0.75	1.4474	1.3284	1.2736	1.2474	0.49	1.4010	1.3284	1.2736	1.2474	1.2474	

T = Sample transmittance (see step 15)

f(T) = Sample correction factor (see step 15)