

UNCLASSIFIED

AD 430724

DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION, ALEXANDRIA, VIRGINIA



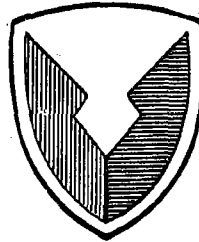
UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

64-9

COATING and CHEMICAL LABORATORY

CATALOGED BY DDC
430724
AS AD NO. _____



CCL REPORT NO. 153

AN ADAPTATION AND EXTENSION OF A QUANTITATIVE INFRA-RED
METHOD FOR THE ESTIMATION OF THE NITROGEN RESIN CONTENT
OF MODIFIED ALKYDS

BY

M. L. ADAMS

AMCMS CODE NO. 5025.11.84205
DA PROJECT 1-H-0-24401-A-110-05

MAR 3 1964

10 JANUARY 1964

ABERDEEN PROVING GROUND
MARYLAND

430724

DESTROY; DO NOT RETURN

DDC AVAILABILITY NOTICE

Qualified requesters may obtain copies of this report from the Defense Documentation Center, Cameron Station, Alexandria, Virginia.

Copies Available at Office of Technical Services, \$ 0.50

THE FINDINGS IN THIS REPORT ARE NOT TO BE CONSTRUED AS AN OFFICIAL DEPARTMENT OF THE ARMY POSITION.

UNCLASSIFIED

Report No. CCL # 153

Copy Number

AN ADAPTATION AND EXTENSION OF A QUANTITATIVE INFRA-RED
METHOD FOR THE ESTIMATION OF THE NITROGEN RESIN CONTENT
OF MODIFIED ALKYDS

By

M. L. Adams

10 January 1964

AMCMS Code No. 5025.11.84205

Dept of the Army Project No.
1-H-0-24401-A-110-05

U.S. ARMY COATING AND CHEMICAL LABORATORY
Aberdeen Proving Ground
Maryland

UNCLASSIFIED

AUTHOR: *M. L. Adams*
M. L. ADAMS, Chemist
Chemical Cleaning &
Corrosion Branch

REVIEWED BY: *M. Rosenfeld*
M. ROSENFELD, Chief
Chemical Cleaning &
Corrosion Branch

APPROVED BY: *C. F. Pickett*
C. F. PICKETT, Technical Director
U.S. Army Coating & Chemical Laboratory

TABLE OF CONTENTS

	<u>Page No.</u>
TITLE PAGE.....	ii
ABSTRACT.....	iii
INTRODUCTION.....	1
DETAILS OF THE METHOD.....	1 - 2
DISCUSSION.....	2
REFERENCES.....	2
APPENDIX A.....	3
Tables.....	4 - 5
APPENDIX B.....	6
Figures.....	7 - 8
DISTRIBUTION LIST.....	9 - 11

ABSTRACT

An adaptation and extension of a previously published infra-red absorbance ratio method for the analysis of vacuum dried films of nitrogen resin modified phthalic alkyd resins is outlined and shows improved accuracy and the inclusion of the determination of benzoguanamine-formaldehyde not previously covered.

I. INTRODUCTION

Federal Specifications TT-E-489c and TT-E-529a for Class B baking alkyd enamels, call for the presence of 10 - 20% of a nitrogen modifying resin. Miller and Shreve (1) developed an infra-red absorbance ratio method for determining urea-formaldehyde and melamine-formaldehyde in such formulations. The results obtained using their method were found to be erratic, particularly for the melamine-formaldehyde content, mainly due to the fact that the 12.25 μ band used for estimating its content is proportionally very weak thus leading to absorbance readings well below 0.1.

In the adaptation outlined here, the NH deformation frequency at 6.43 μ is used for the measurement of melamine-formaldehyde. Since it has been found that one rather than mixtures of nitrogen resins are present, the accuracy of the method also has been improved by basing the standardizations on two component systems rather than three. The Miller and Shreve method has been extended to include the determination of benzoguanamine-formaldehyde which is now also used as the modifying resin.

II. DETAILS OF THE METHOD

A. Standardization

Known mixtures of a short to medium oil length phthalic alkyd resin and each of the nitrogen resins are prepared in which the nitrogen resin content varies from 10 to 20% by weight on the non-volatile basis. Uniform films of the mixtures are spread on rock salt plates and dried for 15 minutes in a 70°C. vacuum oven.

Using a double beam infra-red recording spectrophotometer with sodium chloride optics, quantitative settings, and a rock salt plate in the reference beam, zero and 100% are set at 5.25 μ with the sample in place. The spectrum is then scanned to approximately 7 μ . Base lines are drawn as indicated in Figure 1. For each standard the ratio of the absorbance at 5.76 μ due to the alkyd portion to that due to the nitrogen resin, 6.09 μ for urea-formaldehyde, 6.43 μ for melamine-formaldehyde, and 6.45 μ for benzoguanamine-formaldehyde, is calculated, the absorbance values being those measured from the point of maximum absorption to the base line. Ideally, the thickness of the film should be such that the alkyd absorbance is no greater than 0.6 and the nitrogen resin no less than 0.1. The absorbance ratios obtained for the standards are then plotted against the ratios of the weight percentages on a nonvolatile basis of alkyd to nitrogen resin.

In this work a Perkin-Elmer model 21 infra-red spectrophotometer was used with settings of: resolution, 960; response, 1; suppression, 0; an up scale signal, and a slow scanning speed. The standard curves shown in Figure 2 were obtained from the data recorded in Table 1.

B. Determination of the Nitrogen Resin Content in Unknowns

A thin uniform film of the isolated vehicle is spread on a rock salt plate and carried through the procedure outlined under the standardization. In order to determine the type of nitrogen resin present, after the quantitative scanning from 5.25 to 7 μ is completed, the balance of the spectrum to 15 μ

is scanned rapidly. As indicated in Figure 1, the presence of urea-formaldehyde is indicated by the absorption band at 6.09 μ , melamine-formaldehyde by the band at 12.25 μ , and benzoguanamine formaldehyde by the bands at 12.07 μ and 12.75 μ . The absorbance ratio for alkyd to nitrogen resins is calculated and the concentration ratio, C_R , of alkyd to nitrogen resin determined from the appropriate standard curve.

Calculation

$$\% \text{ nitrogen resin} = \frac{100}{C_R + 1}$$

III. DISCUSSION

Two portions of the infra-red spectra of the three nitrogen resins blended with alkyds are illustrated in Figure 1. Also shown are those bands used for the identification of the nitrogen resin present, the bands used for absorbance measurements, and the positions of the base lines.

Standard curves obtained from the data in Table I are presented in Figure 2.

Table II compares the infra-red results obtained on several qualification samples with those obtained by chemical analysis.

In the Tables and Figures the following abbreviations are used. Absorbance, A and A_R is the ratio of absorbance due to the alkyd to that due to the nitrogen resin; weight percentage concentration, C and C_R is the ratio of percent alkyd to percent nitrogen resin on a nonvolatile basis; alkyd resin, PA; benzoguanamine-formaldehyde, BF; melamine-formaldehyde, MF; urea-formaldehyde, UF.

The infra-red method offers adequate accuracy with a substantial reduction in time of analysis; a sample can be completed in less than half an hour including the drying time. In the event of apparent failures or border line results it may be advisable to apply a chemical determination as a check.

IV. REFERENCES

Miller, C. D. and Shreve, O. D., Anal. Chem. 28, 200-1 (1956).

APPENDIX A

Tables

TABLE I
STANDARDS

I. Alkyd: benzoguanamine-formaldehyde					
A _{5.76}	A _{6.45}	C _{PA}	C _{BF}	A _R	C _R
0.314	0.137	77.2	22.8	2.3	3.4
0.583	0.090	90.8	9.2	6.5	9.9
0.515	0.055	93.7	6.3	9.4	14.9
II. Alkyd: melamine-formaldehyde					
A _{5.76}	A _{6.43}	C _{PA}	C _{MF}	A _R	C _R
0.295	0.086	79.3	20.7	3.4	3.8
0.578	0.112	84.6	15.4	5.15	5.5
0.598	0.055	91.9	8.1	10.9	11.3
III. Alkyd: urea-formaldehyde					
A _{5.76}	A _{6.09}	C _{PA}	C _{UF}	A _R	C _R
0.697	0.180	79.9	20.1	3.9	4.0
0.598	0.111	83.9	16.1	5.4	5.2
0.761	0.102	87.7	12.3	7.5	7.1

TABLE 11

ANALYSIS OF SOME COATINGS

Nitrogen Resin Present	A _R	C _R ¹	% Nitrogen Resin by Infra-red ²	% Nitrogen Resin by Chemical Anal.
BF	3.9	5.9	14	14
BF	4.2	6.4	13.5	13
MF	5.2	5.6	15	15
MF	8.4	8.8	10	10
MF	3.7	4.1	19.5	19
MF	8.1	8.5	10.5	10
UF	5.0	4.8	17	16
UF	7.1	6.6	13	12
UF	9.1	8.3	11	11
UF	7.2	6.7	13	14

1. C_R determined from appropriate standard curve

2. % nitrogen resin $\frac{100}{C_R + 1}$

APPENDIX B

Figures

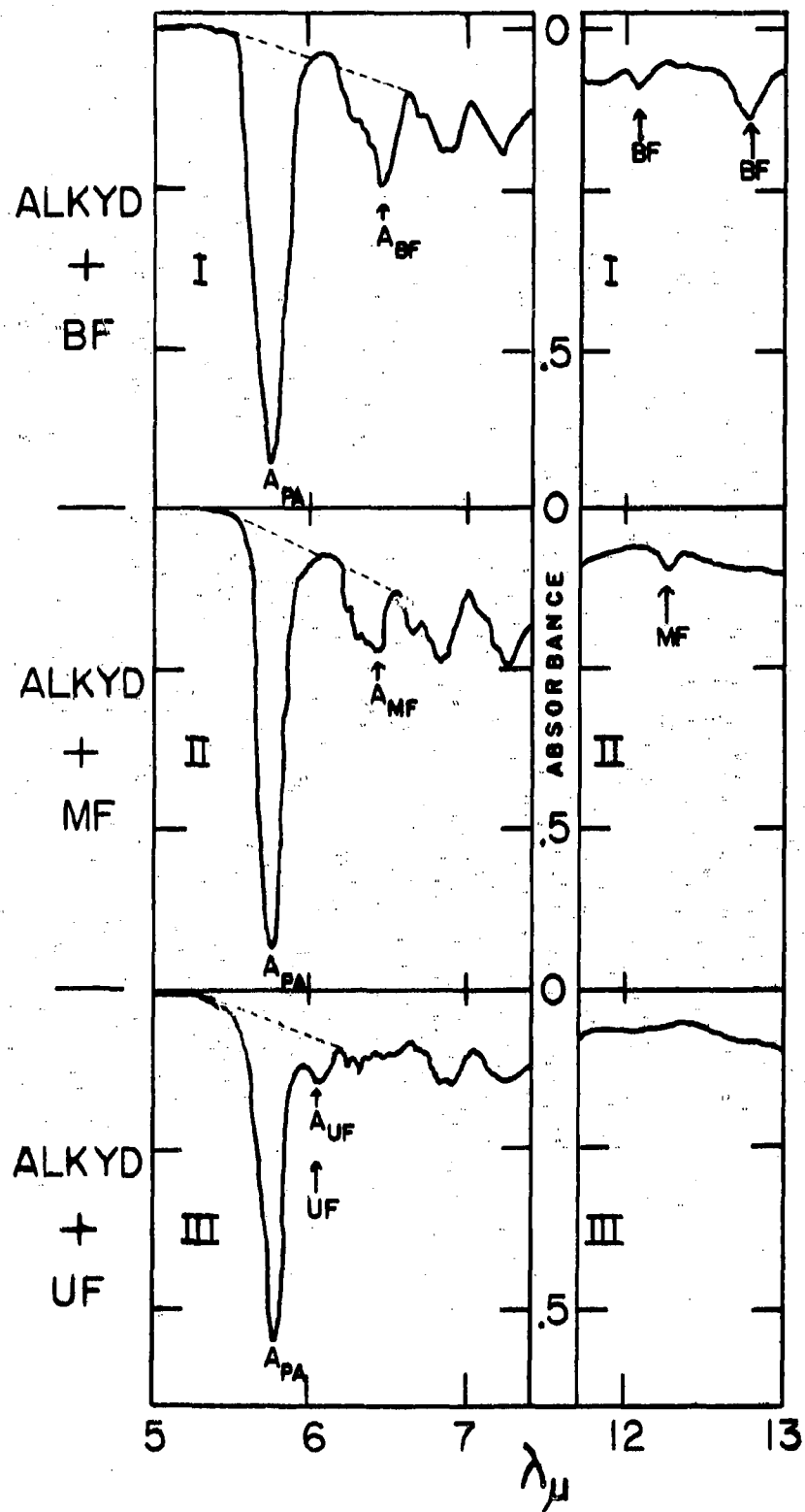


FIGURE 1

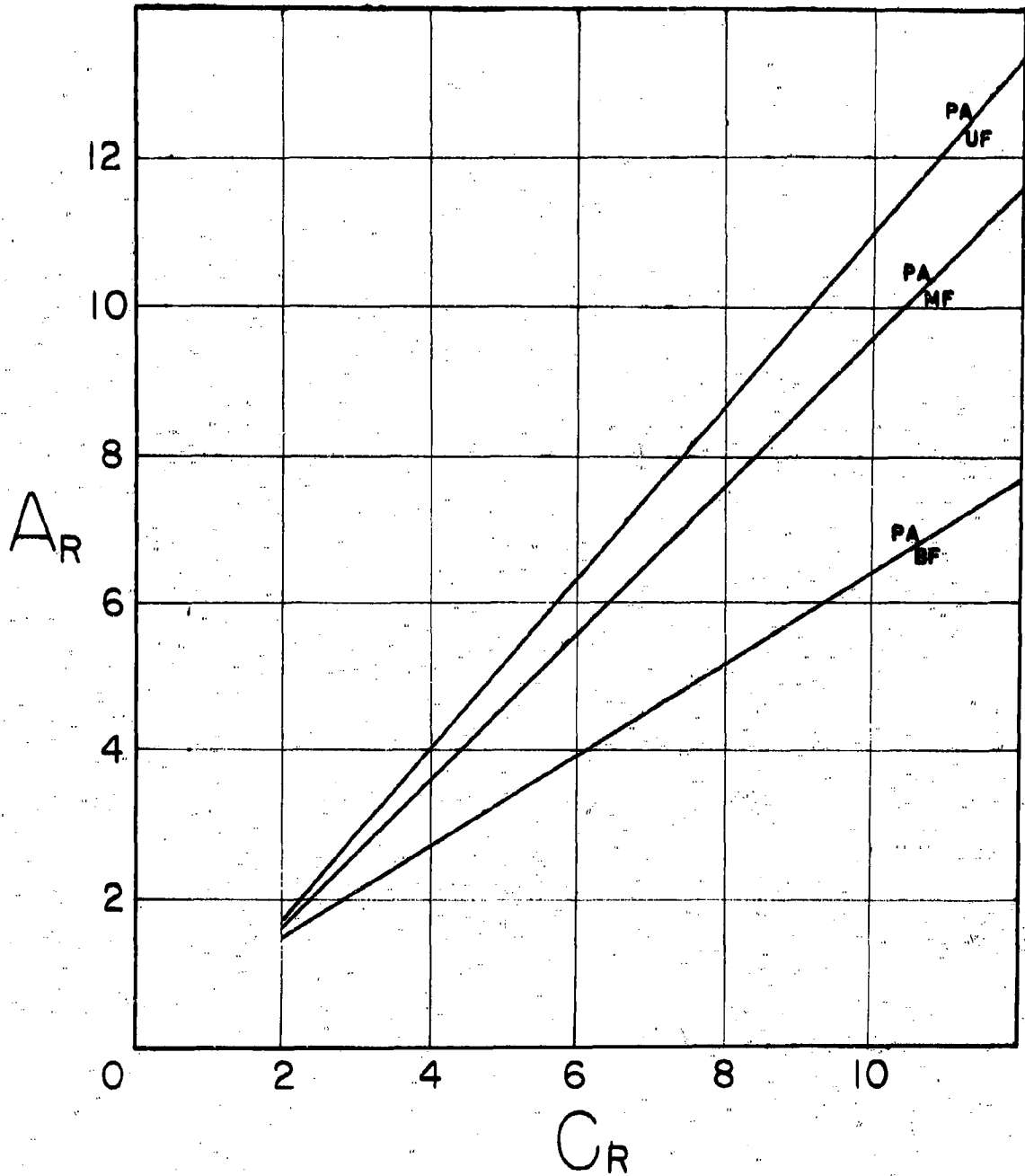


FIGURE 2
STANDARD CURVES

DISTRIBUTION LIST FOR AMCMS CODE NO. 5025.11.84205

<u>Department of Defense</u>	<u>No. of Copies</u>
Defense Documentation Center Cameron Station Alexandria, Virginia	20
<u>Department of the Army - Technical Service</u>	
Commanding General U. S. Army Materiel Command ATTN: AMCRD-RS-CM Washington, D. C. 20315	1
Continental Army Command Department of the Army Fort Monroe, Virginia 23351	3
Commanding Officer U. S. Army Tank Automotive Center ATTN: Mr. J. P. Jones Mr. V. Pogano Warren, Michigan 48090	1 1
Commanding Officer Frankford Arsenal ATTN: SMUFA 1320 Library Philadelphia, Pa. 19137	1 1
Commanding Officer U. S. Army Materials Research Agency Watertown Arsenal ATTN: Technical Information Center Watertown, Mass. 02172	2
Commanding Officer Harry Diamond Laboratory ATTN: Library Washington, D. C. 20425	1
Commanding Officer U. S. Army Test Activity Yuma Test Activity Yuma, Arizona 95364	1
Commanding General U. S. Army Weapons Command ATTN: ANSWE-RDR Rock Island, Illinois 61200	2

DISTRIBUTION LIST CONTINUED

	<u>No. of Copies</u>
Commanding Officer U.S. Army Chemical Research & Development Laboratories ATTN: Librarian Edgewood Arsenal, Maryland 21040	1
Commanding Officer Rock Island Arsenal ATTN: Laboratory 9320 Rock Island, Illinois 61200	1
Commanding Officer Springfield Armory ATTN: Engineering Division Springfield, Mass. 01101	1
Commanding Officer Watervliet Arsenal ATTN: Dr. R. Weigle Watervliet, New York 12189	1
Commanding Officer U.S. Army Munition Command Picatinny Arsenal ATTN: Mr. J. J. Begley Dover, New Jersey 07801	1
Commanding Officer U.S. Army Ballistic Research Laboratories ATTN: Mr. W. C. Pless Mr. R. Elcheberger Mr. J. Sperrazza Aberdeen Proving Ground, Md. 21005	1 1 1
Technical Library Aberdeen Proving Ground, Md. 21005	2
<u>Department of the Navy</u>	
Department of the Navy c/o Navy Liaison Aberdeen Proving Ground, Md. 21005	1
Commander U.S. Naval Weapons Laboratory Dahlgren, Virginia	1
Department of the Navy Chief, Bureau of Naval Weapons Washington, D. C.	1

DISTRIBUTION LIST CONTINUED

Department of the Air Force

No. of Copies

Headquarters

Aeronautical System Division
Air Force Systems Command
ATTN: ASRCE-31
Wright Patterson Air Force Base, Ohio

1

Other Government Agencies

Scientific and Technical Information Facility
ATTN: NASA Representative (S-AK/DL)
P.O. Box 5700
Bethesda, Maryland

3

Office of Technical Services
Acquisitions Section
Department of Commerce
Washington, D. C.

200

Foreign Address

Ministry of Supply Staff
British Joint Services Mission
1800 K Street, N. W.
Washington, D. C.

2

Canadian Army Staff
ATTN: GSO-1, A&R Section
2450 Massachusetts Avenue, N. W.
Washington, D. C.

2

<p>AD No. <u> </u> Accession No. <u> </u> U.S. Army Coating & Chemical Laboratory, Aberdeen Proving Ground, Md., CCL # 153, AN ADAPTATION AND EXTENSION OF A QUANTI- TATIVE INFRA-RED METHOD FOR THE ESTIMA- TION OF THE NITROGEN RESIN CONTENT OF MODIFIED ALKYDS - M. L. Adams - Rpt No. 153 - 10 January 1964, 14 pgs, AMCMS Code No. 5025.11.84205, DA Proj No. 1-H-0-24401-A-110-05</p> <p>An adaptation and extension of a previously published infra-red absor- bance ratio method for the analysis of vacuum dried films of nitrogen resin modified phthalic alkyd resins is out- lined and shows improved accuracy and</p>	<p>Unclassified</p>	<p>Unclassified</p>	<p>AD No. <u> </u> Accession No. <u> </u> U.S. Army Coating & Chemical Laboratory, Aberdeen Proving Ground, Md., CCL # 153, AN ADAPTATION AND EXTENSION OF A QUANTI- TATIVE INFRA-RED METHOD FOR THE ESTIMA- TION OF THE NITROGEN RESIN CONTENT OF MODIFIED ALKYDS - M. L. Adams - Rpt No. 153 - 10 January 1964, 14 pgs, AMCMS Code No. 5025.11.84205, DA Proj No. 1-H-0-24401-A-110-05</p> <p>An adaptation and extension of a previously published infra-red absor- bance ratio method for the analysis of vacuum dried films of nitrogen resin modified phthalic alkyd resins is out- lined and shows improved accuracy and</p>
<p>AD No. <u> </u> Accession No. <u> </u> U.S. Army Coating & Chemical Laboratory, Aberdeen Proving Ground, Md., CCL # 153, AN ADAPTATION AND EXTENSION OF A QUANTI- TATIVE INFRA-RED METHOD FOR THE ESTIMA- TION OF THE NITROGEN RESIN CONTENT OF MODIFIED ALKYDS - M. L. Adams - Rpt No. 153 - 10 January 1964, 14 pgs, AMCMS Code No. 5025.11.84205, DA Proj No. 1-H-0-24401-A-110-05</p> <p>An adaptation and extension of a previously published infra-red absor- bance ratio method for the analysis of vacuum dried films of nitrogen resin modified phthalic alkyd resins is out- lined and shows improved accuracy and</p>	<p>Unclassified</p>	<p>Unclassified</p>	<p>AD No. <u> </u> Accession No. <u> </u> U.S. Army Coating & Chemical Laboratory, Aberdeen Proving Ground, Md., CCL # 153, AN ADAPTATION AND EXTENSION OF A QUANTI- TATIVE INFRA-RED METHOD FOR THE ESTIMA- TION OF THE NITROGEN RESIN CONTENT OF MODIFIED ALKYDS - M. L. Adams - Rpt No. 153 - 10 January 1964, 14 pgs, AMCMS Code No. 5025.11.84205, DA Proj No. 1-H-0-24401-A-110-05</p> <p>An adaptation and extension of a previously published infra-red absor- bance ratio method for the analysis of vacuum dried films of nitrogen resin modified phthalic alkyd resins is out- lined and shows improved accuracy and</p>

<p>Unclassified</p> <p>the inclusion of the determination of benzoguanamine-formaldehyde not previously covered.</p>	<p>Unclassified</p> <p>the inclusion of the determination of benzoguanamine-formaldehyde not previously covered.</p>
<p>Unclassified</p> <p>the inclusion of the determination of benzoguanamine-formaldehyde not previously covered.</p>	<p>Unclassified</p> <p>the inclusion of the determination of benzoguanamine-formaldehyde not previously covered.</p>