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FAILURE OF CAN/CGBS-1.61-95 MARINE ALKYD PAINT TO DRY ON NEOPRENE COATED NYLON SEALING TAPE (U)

by

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Abstract

The failure of CAN/CGSB-1.61-95 marine alkyd paint to dry when applied over a neoprene-coated sealing tape has been investigated. Analysis indicated that the tape and the paint applied to the tape contained several alkyl-substituted phenols. As these compounds are anti-oxidants and the paint cured by an oxidative mechanism, it was postulated that the failure of the paint to cure when applied to the tape was due to the leaching of the alkyl-substituted phenols from the tape into the paint and subsequent inhibition of the oxidative drying mechanism. To test this hypothesis, a commercially available alkyl-substituted phenol, methyl-di-t-butyl phenol, was added to the paint. The paint did not cure, indicating that the alkyl-substituted phenols were responsible for the failure of the paint to cure.

Résumé

L'incapacité qu'a peinture alkyde marine CAN/CGSB-1.61-95 de sécher lorsque appliquée sur un ruban chatterton d'étanchéité revêtu de Néoprène a été étudiée. L'analyse a révélé que le ruban et la peinture appliquée sur lui contenaient plusieurs alkylphénols. Comme ces substances sont des antioxydants, et que le mécanisme de séchage de la peinture fait intervenir une oxydation, une hypothèse fut émise à l'effet que l'incapacité qu'a la peinture de sécher lorsque appliquée sur le ruban vient de la diffusion des alkylphénols du ruban dans la peinture, ce qui empêche ensuite le séchage par oxydation. Pour vérifier cette hypothèse, un alkylphénol commercial, le di-t-butylméthylphénol, fut ajouté à la peinture. Dans ces conditions, la peinture ne séchait pas, ce qui indique que les alkylphénols étaient responsables de l'incapacité de la peinture de sécher.

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Introduction

As part of OPVAL 3/95 - "Radar Absorbing Material (RAM) Installation in Canadian Patrol Frigates", a neoprene coated nylon fabric tape was used to seal the edges of the RAM tiles. The tape, Microsorb Tape 93B, was distributed by Microsorb Technologies, Hopedale, Massachusetts, the same company that supplied the RAM tiles. The tape was bonded to the RAM tiles using a neoprene-based contact cement (3M 1357 Contact Adhesive). Although a good bond between the tiles and the tape resulted, it was reported that tape overcoated with marine alkyd paint (CAN/CGSB-1.61-95[1] or 1-GP-61M) remained tacky. In contrast to this, marine alkyd paint applied over the RAM tile dried. DREA was asked to investigate this phenomenon and comment on the failure of the paint to dry when applied on the neoprene-based tape .

The investigation of the failure of the paint to dry when applied to the tape is described in this report. It was originally thought that the tackiness of the coating on the tape might be to due release of solvent absorbed by either the tape or underlying adhesive during painting. Diffusion of the solvent from the tape or the contact cement into the paint would give the paint an 'undried' character. This was not found to be the case. It was also thought that the lack of drying might be due to the paint itself. 1-GP-61 Marine alkyd paint is a medium oil alkyd that cures by autooxidation. The cure of alkyd paints is dependent upon a number of factors including the correct formulation, that is, the presence of chemicals required for the paint to cure. However, the paint cured when applied to a number of substrates indicating that the formulation was correct. This suggested that the failure of the paint to cure when applied to the tape was due to something in the tape that was affecting the oxidative cure mechanism. Evidence that this was the case is presented in this report.

Experimental

Materials

The RAM tile (Microsorb HP5 Broadband Absorber) and the tape (Microsorb Tape 93B) were received from Microsorb Technologies, Hopedale, Massachusetts. The contact adhesives, one a solvent-based contact cement (3M 1357 Contact Adhesive), and the other a water-based contact cement (3M Neutral 30 High Performance Water Based Contact Adhesive) were received from 3M Canada Limited, London, Ontario. The marine alkyd paint (CGSB 1-GP-61M) was obtained from the Stores System, Formation Halifax.

The di-t-butyl-4-methylphenol (99%, Aldrich Chemical Company, Milwaukee, Wisconsin) was used without further purification. A standard solution containing approximately 1.1 mg/mL di-t-butyl-4-methylphenol in hexane was prepared by dissolving 0.1061g of mg/mL di-t-butyl-4-methylphenol in 100mL of hexane. Samples of 1-GP-61 paint containing approximately 100 ppm, 300 ppm, and 500 ppm di-t-butyl-4-methylphenol were prepared by adding 0.75 mL, 2.19 mL, and 3.97 mL of the standard solution to 7.89 g, 7.32 g, and 7.94 g of the paint respectively.

A 50/50 (V/V) mixture of methylene chloride/hexane was used to extract the neoprene coated nylon tape.

Equipment

Gas chromatography/mass spectrometry (GC/MS) analyses were carried out on a Fisons Model 8000 GC/MS. Mass spectra were acquired in the scanning mode between 25 amu and 500 amu. The GC oven was programmed to hold at 40°C for 5 minutes, ramped at a rate of 10°C/min to 300°C, then held at 300°C for 9 minutes. Total run time was 40 minutes. Helium was used as the carrier gas.

Pyrolysis was carried out on a Chemical Data System Model 122 pyroprobe using a platinum coil probe and 25 mm quartz pyrolysis tubes. The sample was heated at the maximum rate (temperature ramp off) to 700°C using a 20 second interval time.

Results and Discussion

Background

Paints

A paint consists of three major components, the binder, the pigment, and the solvent. The binder acts to hold the other components of the paint film together, the pigment to give the film its desired color and hiding properties and enhance hardness and weathering characteristics, and the solvent to dissolve the semisolid binders so that they can be applied to the surface to be coated or to act as a diluent in a free-flowing coating and improve application characteristics[2]. In addition to the major components, a paint can include additives such as plasticizers, driers, wetting agents, and emulsifiers.

The binder is the most important component of a paint and many of the properties of a paint, such as its mode of drying (cure), mechanical properties, and adhesion depend on the binder., As many binders are inherently brittle and have poor adhesion, plasticizers are added to paint formulations to improve film flexibility. Plasticizers must be non-volatile so that they remain in the film following drying, or they can be incorporated into the backbone of the polymer that forms the binder.

Cure or Drying

Although there are a large number of paint formulations, they all dry and form a film in one of two ways. These are physical film formation, which results from the evaporation of solvent, and chemical film formation, which involves a chemical reaction. Lacquers are examples of coatings that cure by physical film formation, that is, evaporation of solvent, while alkyds and epoxies are examples of paints that dry through chemical film formation.

Chemical film formation can be subdivided into oxidative drying and curing. The first step in the oxidative drying is the uptake of oxygen from the air. This is followed by an oxidation step that results in the cross-linking of the resin portion of the coating. In curing, the formation of chemical bonds does not depend on the uptake of oxygen. Alkyd paints cross-link through an oxidative drying reaction while epoxy-based paints dry through a chemical reaction.

The rate of drying and the development of the properties of a paint film will depend upon a number of factors. These can include temperature, the availability of oxygen, the effectiveness of catalysts or driers that increase the rate of oxygen uptake and subsequent cross-linking reactions, and the reactivity of the components in coatings that cure chemically. Storage temperature and storage time can also affect the quality of film that can be expected from a paint formulation, as can the nature of the pigments and additives, such as plasticizers, used in the paint.

Drying Oil Alkyds.

1-GP-61 Marine alkyd paint is a drying oil alkyd and as such cures by an oxidative mechanism. It has been shown that the autooxidation of alkyds containing drying and semidrying oils proceeds through at least four steps[3]. These are: 1) the destruction of antioxidants in the paint film, 2) the uptake of oxygen and the formation of peroxides and conjugated double bonds, 3) the acceleration of the decomposition of the hydroperoxides to form radicals to the point where the reaction becomes autocatalytic, and 4) the start of the polymerization and cleavage reactions to form the final paint film.

Initiation of the autooxidation process involves the reaction of atmospheric oxygen with hydrogen atoms alpha to the double bonds in the drying oils to form hydroperoxides. This subsequently leads to isomerization of the double bonds in the oils, polymerization, degradation and the formation of oxidation products. This process is shown in Scheme 1[4].

Scheme 1

Initiation $$RH + O_2 \rightarrow R^{\bullet} + {}^{\bullet}OOH$$

Propagation $$R^{\bullet} + O_2 \rightarrow ROO^{\bullet}$$

$$ROO^{\bullet} + RH \rightarrow ROOH + R^{\bullet}$$

Termination	$ROO^{\bullet}$	+	$R^{\bullet}$	→	ROOR		
	$ROO^{\bullet}$	+	$ROO^{\bullet}$	→	ROOR	+	O_2
	$R^{\bullet}$	+	$R^{\bullet}$	→	RR		

The reaction of the various drying oils used in alkyd paints has been found to depend on a number of factors including the number of double bonds in the oil, whether or not the double bonds are conjugated, and the arrangement of substituents around the double bonds. When the oxygen concentration in the paint film is low, propagation terminates quickly resulting in a low molecule weight polymer.

Metallic salts that catalyze the drying reaction can be used to decrease the cure time of alkyds containing drying oils. To act as drying catalysts the metal ions should undergo oxidation from a lower to a higher valence state when exposed to the fatty acid hydroperoxides in the coating. These catalysts, or driers, are commonly hydrocarbon soluble salts of metals such as cobalt, manganese, lead, iron, calcium, zinc and aluminum.

Chemicals capable of forming complexes with the metal ions in driers have been found to adversely affect the efficiency of driers[5]. That is, the interaction between the metal ions and the complexing chemicals prevents the metal ion from catalyzing the autooxidation of the drying oils in the paint. Other chemicals, such as antioxidants, can act to inhibit or prevent the autooxidation reaction that leads to the drying of alkyd paints. These work by interrupting the chain reaction that is responsible for the drying of the paints. Hindered phenols are examples of antioxidants that work by donating electrons and breaking the chain reaction responsible for the drying of the paint[6].

Definition of the Problem

Tests indicated that the paint would dry when in contact with a number of substrates including a glass slide, the exterior of a RAM tile, and the adhesive used to attach the tape to the RAM tile. However, it did not cure when applied to the tape. This suggested that something in the tape was affecting the reactions leading to the drying of the paint. Our approach was to analyze samples of the tape and the paint before and after application to the tape to determine if something in the tape could be linked with the failure of the paint to dry.

Analysis of the Neoprene Tape and Undried Paint

A pyrogram, that is, the chromatogram resulting from pyrolytic degradation at 700°C, of a sample of the methylene chloride/hexane extract of neoprene-coated nylon tape is shown in Figure 1a. The peaks in the pyrogram are low molecular weight compounds, such as additives, extracted from the rubber portion of the tape or their pyrolytic degradation products. For instance, compounds with the molecular formula C_8H_{16} gave rise to the peaks with retention times between 5 and 6 minutes, an eight carbon alcohol the peak at 7.04 minutes, phenol the peak at 11.72 minutes, and chloromethylheptane the peak at 12.03 minutes. Phthalic and phosphoric acid esters gave rise to the majority of peaks with retention times of greater than 30 minutes. These compounds included dioctylphthalates and 2-ethylhexyl-diphenylphosphate (30.25 minutes), a common phosphate ester-based plasticizer.

Pyrograms of 1-GP-61 alkyd paint and a sample of 1-GP-61 alkyd paint that had been applied to the neoprene coated nylon tape are shown in Figures 1b and 1c respectively. The major degradation product of the alkyd paint was phthalic anhydride (1,2-benzenedicarboxylic acid anhydride) at 17.72 minutes. Other products included benzoic acid, hexadecanoic acid (25.53 minutes), octadecenoic acid (27.38 minutes), octadecanoic acid (27.58 minutes) and a phthalic acid ester (31.05 minutes). These degradation products are consistent with an alkyd resin prepared from phthalic acid, a polyol, and fatty acids. These compounds were also present in the pyrogram of the sample of paint removed from the tape.

However, comparison of the pyrograms indicated that the pyrogram of the paint applied to the tape had a number of peaks not found in the pyrogram of the paint sample. Comparison of Figures 1a and 1c indicated that many of these peaks corresponded to those in the pyrogram of the tape extract. For example, the phosphate ester and the compounds with retention times greater than 30 minutes were found in both pyrograms.

These results strongly suggested that something in the tape extract was preventing the paint from drying. To confirm this, the extract from the tape (10% by weight) was added to a sample of the paint. The coating did not dry. Similar results were observed when 4% by weight of the tape extract was added to the paint. Pyrograms of the undried paint samples with 10% and 4% tape extract are shown in Figures 2a and 2b respectively. Comparison of these pyrograms and the pyrogram of the undried paint taken from the surface of the tape (Figure 1c) indicated that they were similar. For instance, ethylhexyldiphenylphosphate was found in all three pyrograms, at 30.14 minutes in Figure 1c, at 30.36 minutes in Figure 2a, and at 30.24 minutes in Figure 2b.

Analysis of the Tape Extract

As something in the tape extract was affecting the drying of the paint, a thorough analysis of the tape extract was carried out. A chromatogram of a sample of the tape extract is shown in Figure 3. The compounds found in the chromatogram of the extract are not exactly the same as those found in the pyrogram of the same material. This is the result of heating and subsequent thermal degradation of some of the components of the extract during pyrolysis.

The difference in the retention times of the same compounds in Figures 1a and 3 resulted from differences in the conditions for the chromatographic separation of the compounds following either pyrolysis or injection of the sample. The GC was operated in the pressure control mode for pyrolysis GC/MS, and in the flow control mode for conventional GC/MS. In the pressure control mode, the pressure at the head of the column is constant throughout the run. Carrier gas flow through a column at constant head pressure is dependent on temperature. As the viscosity of a gas increases with temperature, the flow decreases during a GC run in which the column temperature is increased. In the flow control mode a flow rate is maintained throughout the run. This is accomplished by increasing the pressure at the head of the column as the temperature increases.

The major constituents of the tape extract were similar to those found in the pyrolyzed tape sample and the paint applied to the tape. For instance, the phosphate ester plasticizers are major components in all three chromatographic traces.

Analysis of the chromatogram of the tape extract indicated that there were a large number of compounds present in low concentrations in the tape extract. The chromatogram of the tape extract between 17.25 minutes and 20.00 minutes is shown in Figure 3b. Some of these compounds, including diisopropylphenol (18.02 minutes), methyldiisopropylphenol (18.53 minutes), and dibutylethylphenol (19.92 minutes), were alkyl substituted phenols. Alkyl substituted phenols (or hindered phenols) are common antioxidants and could act to inhibit the oxidative cross-linking (drying) of the alkyd-based paint.

Closer inspection of the pyrograms of the paint applied to the tape and the paint containing the tape extract also showed that alkyl substituted phenols were present in these samples. The pyrograms between 18.25 minutes and 21.00 minutes are shown in Figures 4a and 4b along with the traces for the extracted ions 234, 219, 192, and 177. The ions 234 and 219 are characteristic of dibutylethylphenol and the ions 192 and 177 are characteristic of methyldiisopropylphenol. Dibutylethylphenol (20.98 minutes and 20.95 minutes) and methyldiisopropylphenol (19.52 minutes and 19.46 minutes) were found in both samples. In contrast to this, the pyrogram of 1-GP-61 paint did not contain either of these compounds. These results suggested that antioxidants (alkyl substituted phenols) present in the tape were interrupting the oxidative cure mechanism of the alkyd paint.

To confirm that alkyl substituted phenols would prevent 1-GP-61 paint from drying, samples of 1-GP-61 paint containing approximately 100 ppm, 300 ppm, and 500 ppm di-t-butyl-4-methylphenol were prepared. The sample containing 100 ppm di-t-butyl-4-methylphenol dried after 2 days, while the samples containing 300 ppm and 500 ppm di-t-butyl-4-methylphenol were still tacky after 5 days. The control sample, that is, the paint without any added antioxidants, dried overnight.

Conclusions

The 1-GP-61 paint did not cure as the result of antioxidants retarding the oxidative curing mechanism. The antioxidants were leached from the neoprene-based sealing tape by the solvent in the paint.

If this tape is used to seal radar absorbing material tiles, it should be coated with a paint that does not cure by an oxidative mechanism, such as an epoxy or a polyurethane coating. Alternatively, the manufacturer of the tape can color match the tape to the topside gray used on Canadian Forces ships.

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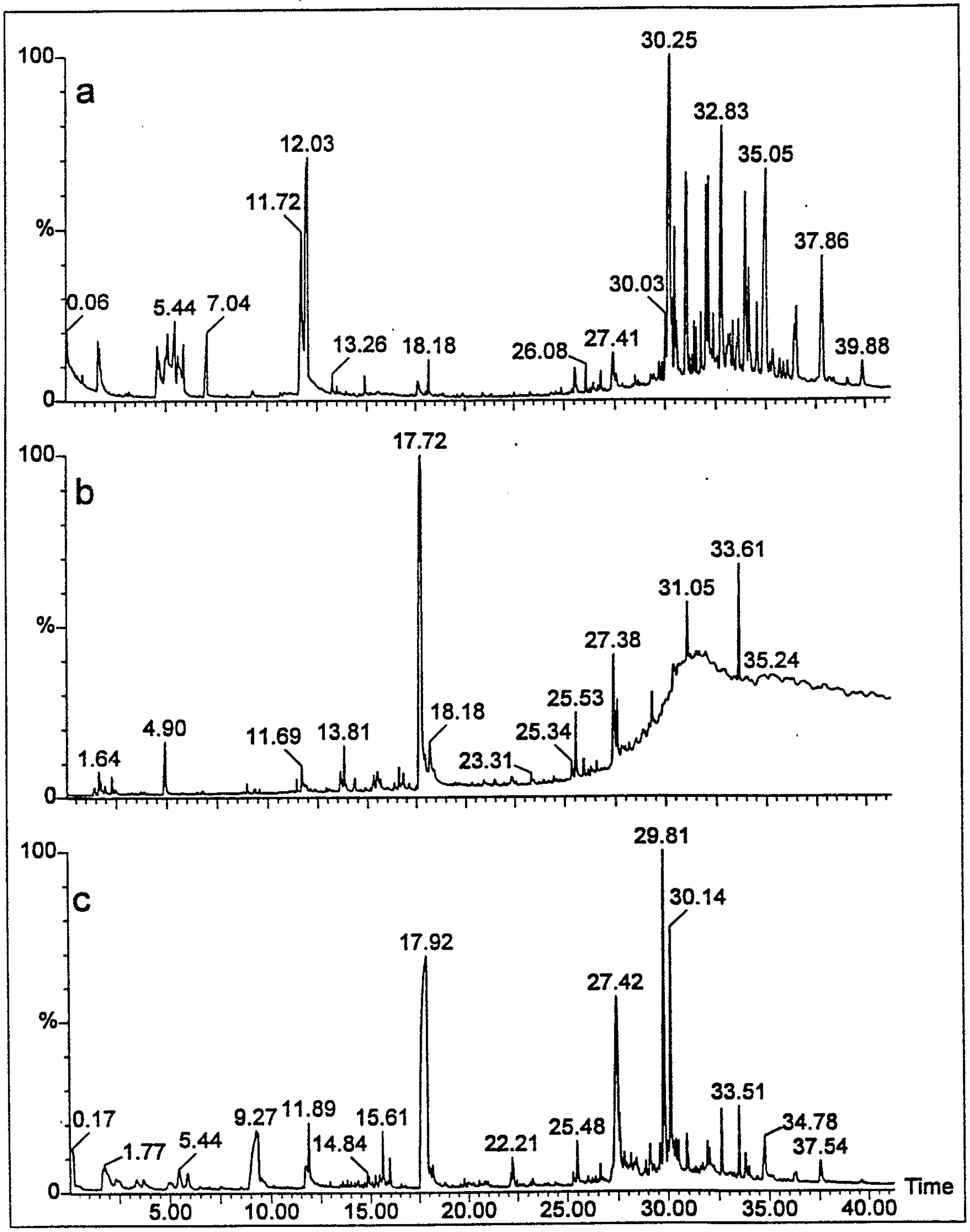

Figure 1 - Pyrograms of samples of a)neoprene-based tape extract, b) CAN/CGSB-1.61-95 marine alkyd paint, and c) CAN/CGSB-1.61-95 marine alkyd paint applied over neoprene-based paint.

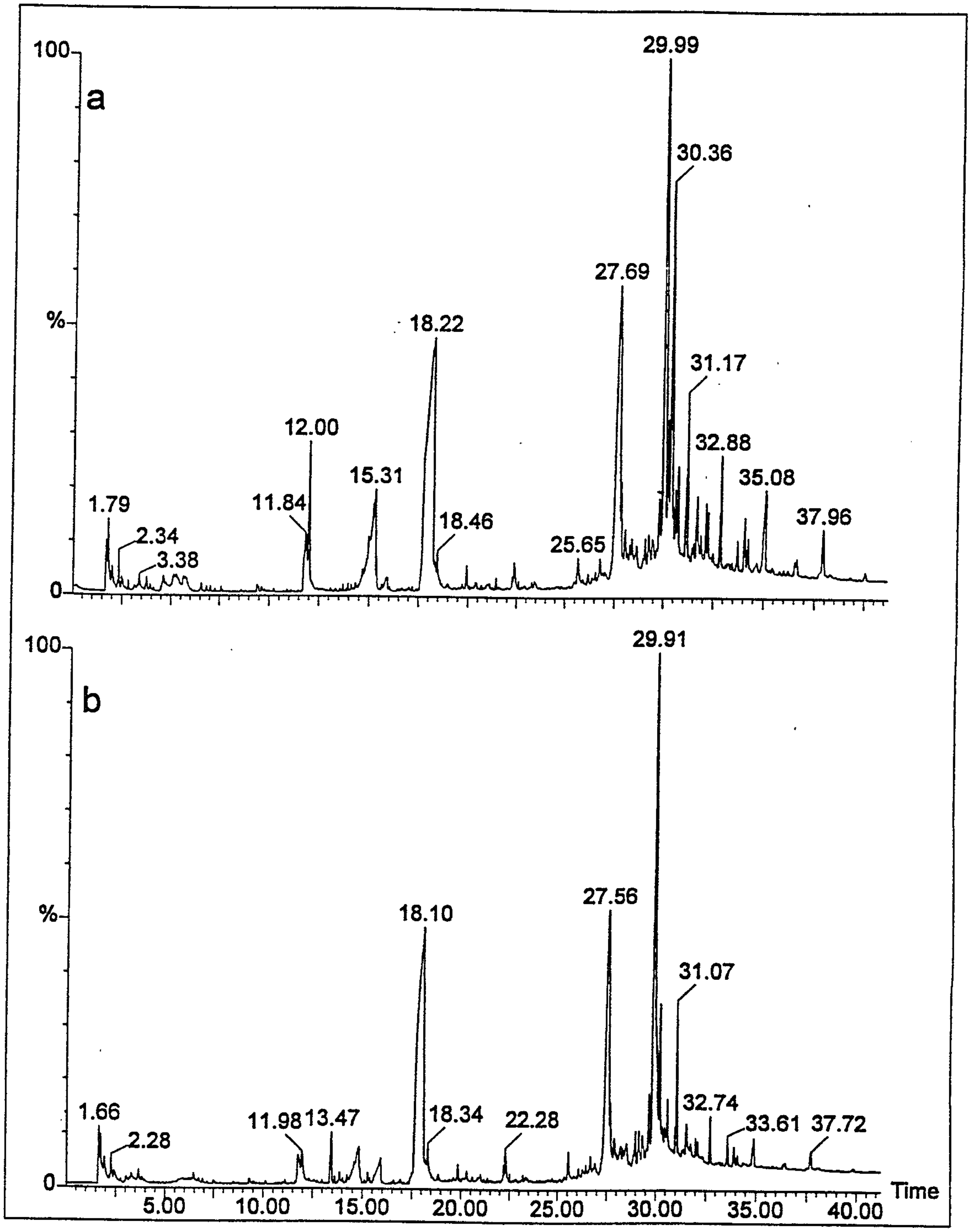

Figure 2 - Pyrograms of samples of CAN/CGSB-1.61-95 marine alkyd paint containing a) 10% and b) 4% by weight neoprene-based tape extract.

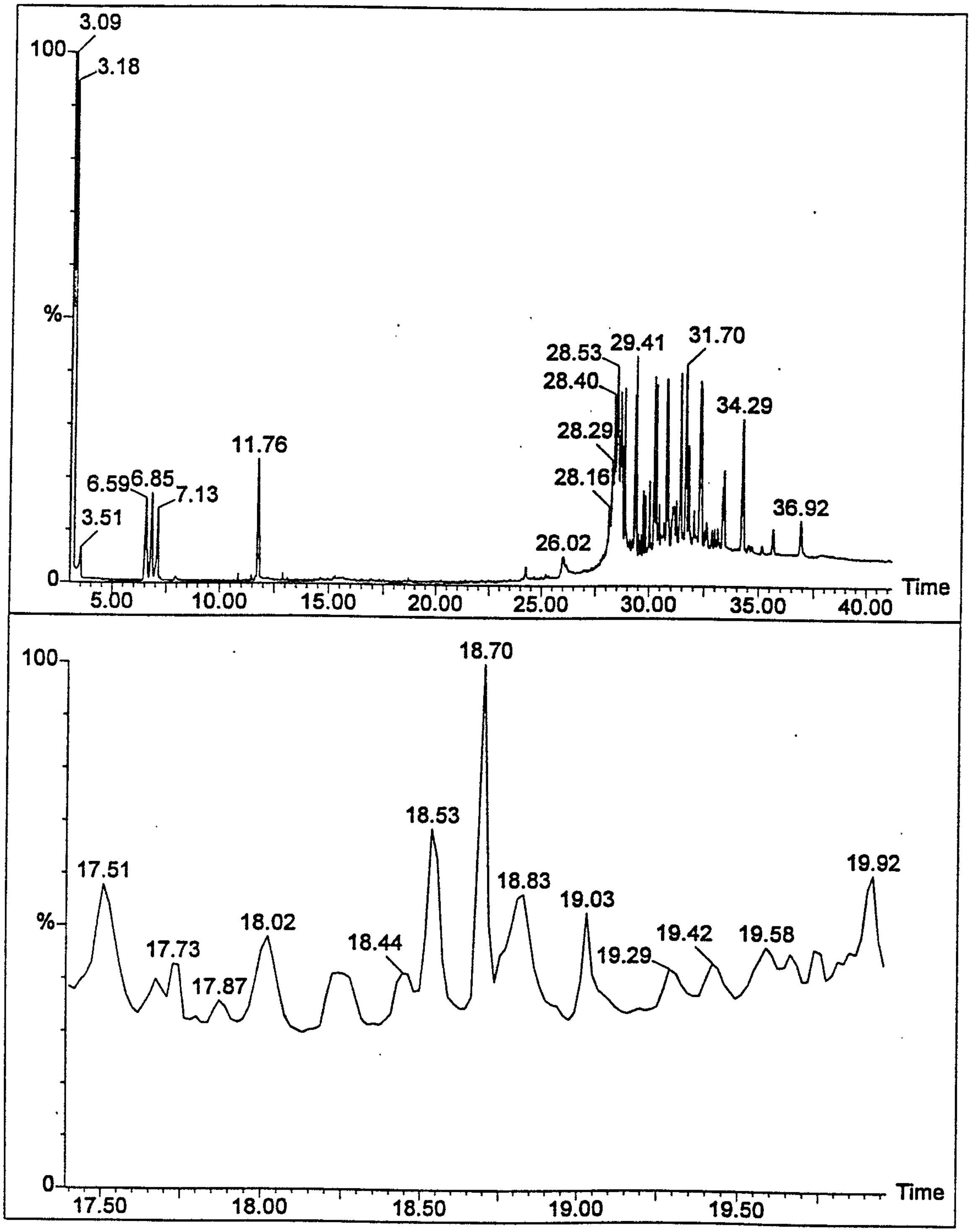

Figure 3 - a) Chromatogram of a sample of extract from the neoprene-based tape and b) chromatogram of the extract between 17.25 minutes and 20.00 minutes.

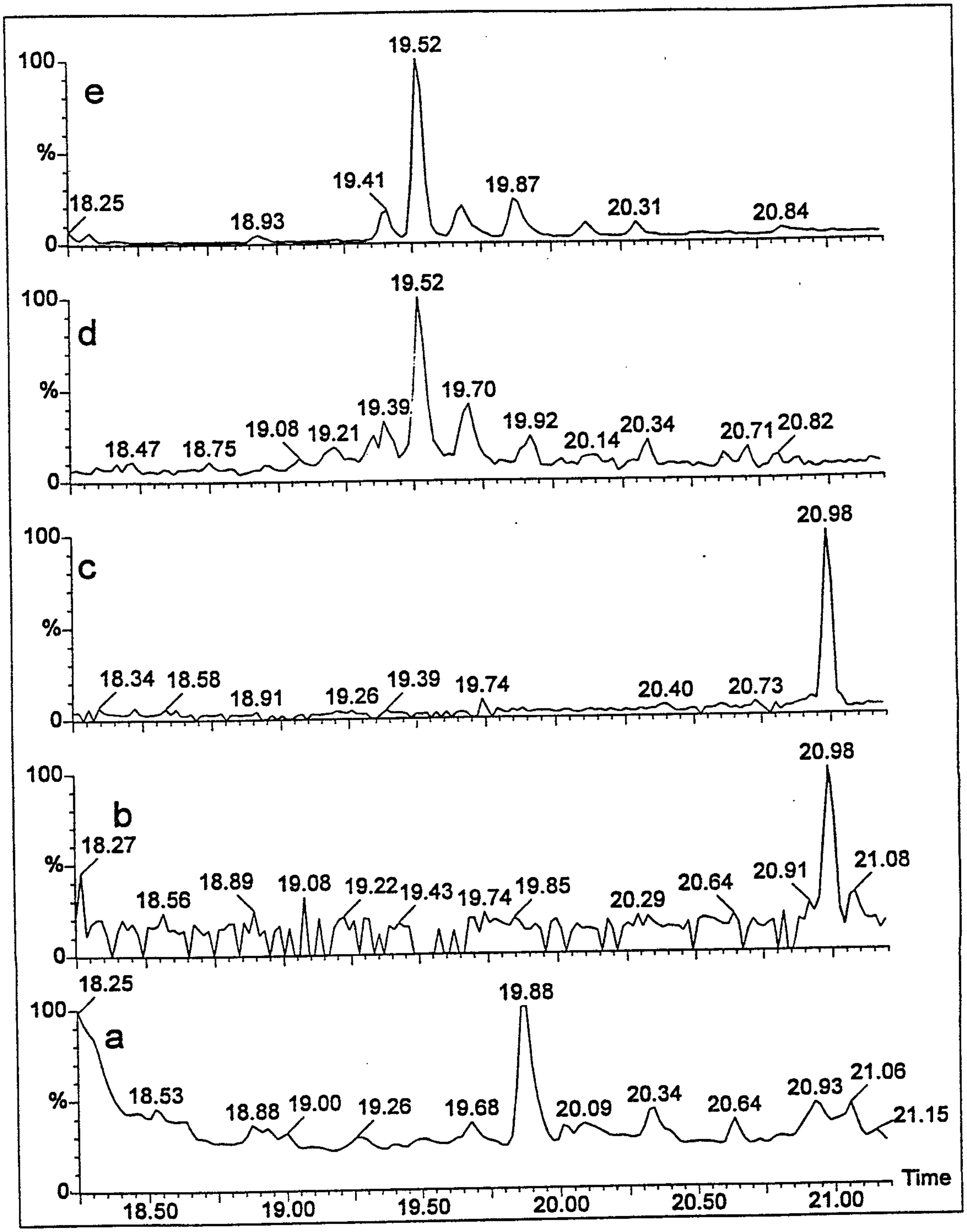

Figure 4 - a) Pyrogram of a sample of alkyd paint applied to the neoprene-based tape between 18.25 and 21.00 minutes. Traces of extracted ions of mass b) 234, c) 219, d) 192 and e)177. Ions of mass 234 and 219 are characteristic of ethyl-di-t-butylphenol and ions of mass 192 and 177 are characteristic of diisopropylphenol.

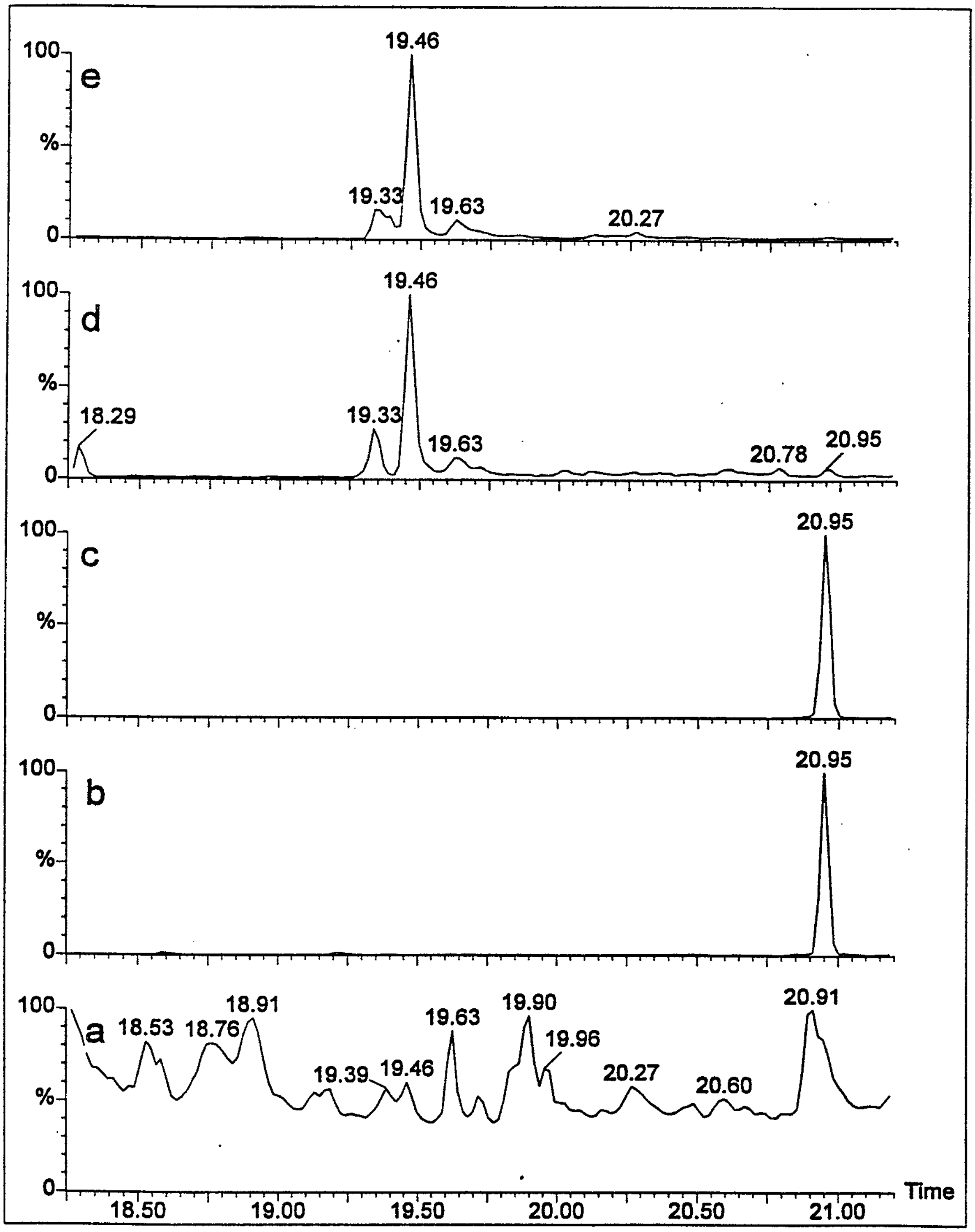

Figure 5 - a) Pyrogram of a sample of alkyd paint containing 4% tape extract between 18.25 and 21.00 minutes. Traces of extracted ions of mass b) 234, c) 219, d) 192 and e)177. Ions of mass 234 and 219 are characteristic of ethyl-di-t-butylphenol and ions of mass 192 and 177 are characteristic of diisopropylphenol

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The failure of CAN/CGSB-1.61-95 marine alkyd paint to dry when applied over a neoprene-coated sealing tape has been investigated. Analysis indicated that the tape and the paint applied to the tape contained several alkyl-substituted phenols. As these compounds are anti-oxidants and the paint cured by an oxidative mechanism, it was postulated that the failure of the paint to cure when applied to the tape was due to the leaching of the alkyl-substituted phenols from the tape into the paint and subsequent inhibition of the oxidative drying mechanism. To test this hypothesis, a commercially available alkyl-substituted phenol, methyl-di-t-butyl phenol, was added to the paint. The paint did not cure, indicating that the the alkyl-substituted phenols were responsible for the failure of the paint to cure.

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failure
antioxidants
alkyl substituted phenols

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