

CHROM. 10,142

Note

Identification and determination of 1,3-diisobutyrate-2,2,4-trimethylpentane (Texanol isobutyrate) in polyvinyl chloride plastisol coating formulations

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(Received March 18th, 1977)

Analytical problems may arise in the quality control of the manufacture of plastisol films with desired durability and resistance to staining by foot traffic due to the necessarily low levels of plasticisers in the final products and also from the differential loss of certain components of plasticiser blends during processing. A mixture of Texanol isobutyrate (TXIB), butyl benzyl phthalate (BBP), and an epoxy-dised tall oil (ETO) have been used in the manufacture of a fused plastisol film from a paste polyvinyl chloride resin. The addition of TXIB was to attain a suitable plastisol spreading viscosity, the BBP was the primary plasticiser¹ and ETO a heat stabiliser to protect the plastisol from degradation during fusion. Because of the high fusion temperature, 200°, and the large exposed surface area to volume ratio (film thickness 0.016 in.) much of the volatile plasticiser components are lost, particularly TXIB, the residual amount of which is important in final product performance.

EXPERIMENTAL

Materials and formulations

The plasticisers used herein are specified in Table I. The solvent used, tetrahydrofuran, was of technical reagent grade (BDH, Poole, Great Britain). The dibutyl phthalate, internal standard, was supplied by Geigy (Manchester, Great Britain). The paste resin was Breon P 130/1² (BP, London, Great Britain).

Plastisol formulations were prepared in the usual manner by mixing components under vacuum, to remove air, in a paddle type blender. A typical formulation composition is shown in Table II.

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TABLE I
PLASTICISERS

No.	Name	Abbreviation code	Source
1	Texanol isobutyrate	TXIB	Eastman (Hemel Hempstead, Great Britain)
2	Butyl benzyl phthalate	BBP	Monsanto (Ruabon, Great Britain)
3	Epoxydised tall oil	ETO	Lankro (Manchester, Great Britain)

TABLE II
TYPICAL FORMULATION COMPOSITION BEFORE FUSION

Component	Percentage by weight
Paste polymer	71.7
TXIB	17.9
BBP	3.6
EPO	3.6
Other components	3.12

Apparatus

A Becker Model 417 gas chromatograph equipped with a flame ionisation detector was used isothermally. The optimised conditions for analysis, determined in preliminary studies were: column 1 m \times 2.6 mm I.D. stainless steel; stationary phase, 10% SE-30 on Chromosorb W HP (80–100 mesh); oven temperature, 200°; injection block temperature, 250°; detector temperature, 275°; air flow-rate 300 ml/min; hydrogen flow-rate, 30 ml/min; carrier gas, nitrogen; nitrogen flow-rate, 30 ml/min; recorder, 10 mV; attenuation, \times 8.

Preparation of sample solutions

TXIB calibration standards were 0.025–0.10% (w/v) in tetrahydrofuran containing 0.2% (w/v) dibutyl phthalate. BBP and EPO solutions were 0.1% (w/v) in tetrahydrofuran containing 0.2% (w/v) dibutyl phthalate.

The finished flooring products normally consist of several layers of different materials. The upper pastisol layer, the wear resistant layer, thus has to be carefully pared from the flooring base layer composition to avoid contamination. The separated upper layer (0.2 g) was cut into small pieces and dissolved in tetrahydrofuran (10 ml) containing 0.2% (w/v) dibutyl phthalate. Gentle warming facilitates the dissolution. 3.5- μ l volumes of each sample solution, in turn, were injected directly on column and chromatograms recorded.

RESULTS

It was found that only TXIB, BBP, the internal standard and solvent eluted from the column and were resolved well under the conditions specified. ETO and the other components remained on column. The retention data are given in Table III.

The calibration graph for TXIB was linear over the range examined. Chromatograms of dissolved plastisol films showed only peaks for TXIB and that BBP was absent from the fused materials examined as shown in Fig. 1. Quantitative examination based on peak heights of 5 replicate injections from single samples gave

TABLE III
RETENTION TIMES

Component	Time (min)
Tetrahydrofuran	0.1
TXIB	0.83
Dibutylphthalate	4.13
BBP	5.1
ETO	retained on column

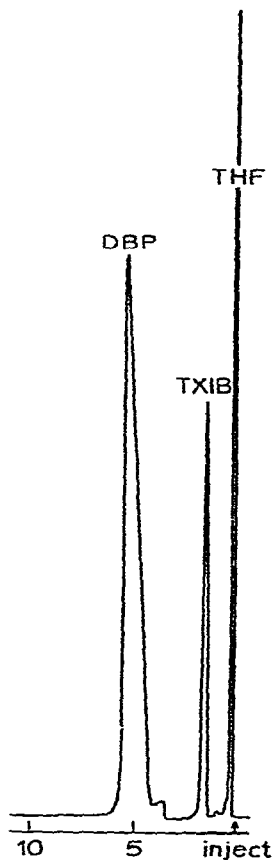


Fig. 1. Typical chromatogram of fused polyvinyl chloride plastisol coating. DBP = dibutyl phthalate; THF = tetrahydrofuran.

standard deviations of 0.11% TXIB at levels which were for each sample studied in the range 1.3–1.6% TXIB.

DISCUSSION

The differential loss of plasticisers during the fusion stage of the manufacturing process was confirmed by infrared (IR) examination of non-volatile portions of

extracts of fused plastisol films. Extraction (24 h) of fused coating³ in ethanol (94 vol %) gave a non-volatile oily residue (105°) of 6.5% (w/w). The initial plasticiser plus oil content was 28.2% thus some 21.7% was lost on fusion.

IR examination of the extract showed it to be mainly ETO (b.p. 260° at 2 mm Hg). The peaks between 650 cm⁻¹ and 800 cm⁻¹, characteristic of BBP (b.p. 370° at 768 mm Hg), were absent and those for TXIB were weak.

The proposed method is more rapid than solvent extraction and/or hydrolysis⁴ procedures and allows ready quantitation of the residual volatile plasticiser, TXIB, and is thus suitable for routine quality control in the manufacture of plastisol based products.

ACKNOWLEDGEMENT

We are most grateful to J. Halstead Ltd. for the preparation of materials and for the provision of experimental facilities for one of us (P.S.).

REFERENCES

- 1 D. Thorburn Burns, W. P. Hayes and P. Steele, *J. Chromatogr.*, 103 (1975) 241.
- 2 *Breon P 130/1 Paste Resin*, Technical Manual No. 2, B.P. Chemicals Ltd., 1957.
- 3 L. H. Ruddle, S. D. Swift, J. Udris and P. E. Arnold, *Proc. Soc. Anal. Chem. Conf., Nottingham, 1965*, Heffer, Cambridge, 1965, p. 224.
- 4 J. Haslam, W. Soppelt and H. A. Willis, *Appl. Chem.*, 1 (1951) 112.