

## THERMODIFFUSIVE CHARACTERISTICS OF SOME ADHESIVE, FILM-FORMING MATERIALS AND THE COMPONENT SOLVENTS

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The TG curves obtained for various temperatures and evaporation times allowed establishment of the thermodiffusive characteristics of the solvents frequently used in adhesive and film-forming materials. Determinations were carried out in air with a flow rate of 5 l/h, on pure solvents, on mixtures with solvent ratios corresponding to those in the final products, and on the final products. The temperature range of the study was 17–35°, the evaporation period for most cases at each temperature being 25 minutes. The method leads to the evaporation rates of such solvents with an average standard deviation of 4 per cent.

The evaporation rate is a value used to establish solvent concentrations in industrial workshops in order to avoid the attainment of inflammability and toxicity limits.

In the literature [1–3], the evaporation rate is given as a value relative to that for ethyl ether or butyl acetate, in the form of a volatility index at 20°.

In the present paper, the evaporation rate is determined as a conventional value for the main solvents used in film-forming and adhesive materials.

The study was performed on pure solvents, on mixtures of solvents with compositions corresponding to the compositions of the groups of solvents currently used in the production of some adhesives based on neoprene rubber (Prenadez) and on acetals, as well as on the materials as such.

### Experimental

The solvents tested were acetone, methyl ethyl ketone, trichloroethylene, ethyl acetate, butyl acetate, methyl alcohol, ethyl alcohol, isobutyl alcohol, benzene, toluene, p-xylene and water.

To establish the evaporation rate, the weight of substance evaporated was determined by means of a Paulik–Paulik–Erdey derivatograph equipped with a gas titrimeter.

For the assays, a cylindrical crucible with  $\phi = 20.3$  mm and  $h = 22.3$  mm was used.

For most assays, the evaporation time was 25 minutes and the studied temperature range was between 17 and 35°; the temperature was kept constant within  $\pm 0.5^\circ$ . The sensitivities used in tracing the TG curves ranged between 50 and 500 mg. The determinations were carried out under dynamic conditions, with an air velocity of 0.5 m/s

over the assay surface, the flow rate of the driven and absorbed air being 5 l/h. Dynamic conditions were used in order to simulate actual evaporation conditions; the values of the evaporation rates determined under static conditions are lower by 8 per cent than those obtained under dynamic conditions.

To determine whether an additivity law is observed or not during the release of the component solvents of adhesive and film-forming materials, a Carlo-Erba gas-chromatograph was used with a detection system of catharometer type, using as stationary phase bis-2-methoxy ethyl adipate deposited on silanized Chromosorb W. The assays involved the absorption of the released solvents in acetone.

### Results and discussions

The experimental results obtained are illustrated in Table 1 and Figs 1–6.

For the pure solvents studied, except benzene, the evaporation rate increases linearly with temperature in the range 17–35°.

For benzene and technical solvents (extraction petrol and ethyl acetate) the evaporation rate no longer varies linearly with temperature. This demonstrates a different dependence of the intermolecular forces on temperature.

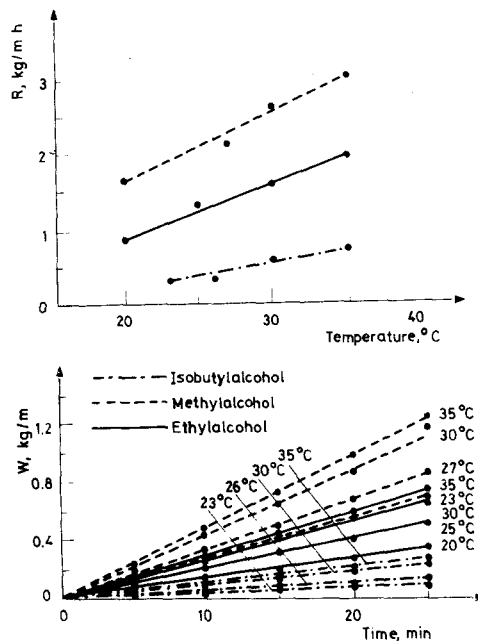


Fig. 1 Evaporation rates of methyl, ethyl and isobutyl alcohols as functions of temperature and time

**Table 1** Evaporation rates of studied solvents

Solvent	Temp., °C	kg/m <sup>2</sup> h	kg/m <sup>2</sup>				
			5'	10'	15'	20'	25'
Water	20	0.13	0.01	0.02	0.04	0.05	0.06
	26	0.22	0.02	0.04	0.05	0.07	0.09
	30	0.29	0.03	0.05	0.07	0.10	0.12
	35	0.36	0.03	0.06	0.09	0.12	0.15
Isobutyl alcohol	23	0.30	0.03	0.06	0.08	0.10	0.12
	26	0.33	0.03	0.06	0.09	0.11	0.14
	30	0.57	0.05	0.09	0.15	0.19	0.24
p-Xylene	35	0.70	0.06	0.11	0.18	0.23	0.29
	23	0.37	0.03	0.06	0.09	0.12	0.15
	26	0.47	0.04	0.07	0.11	0.15	0.19
	30	0.67	0.05	0.11	0.16	0.22	0.28
Toluene	35	0.86	0.06	0.14	0.21	0.28	0.36
	18	0.39	0.03	0.07	0.10	0.14	0.17
	26	0.62	0.06	0.12	0.16	0.22	0.28
	30	0.80	0.06	0.12	0.18	0.24	0.31
Ethyl alcohol	35	1.03	0.09	0.18	0.27	0.37	0.47
	20	0.83	0.07	0.15	0.22	0.28	0.34
	25	1.27	0.11	0.22	0.33	0.43	0.53
	30	1.55	0.14	0.29	0.42	0.56	0.69
Methyl alcohol	35	1.83	0.17	0.33	0.48	0.62	0.76
	23	1.62	0.16	0.29	0.43	0.56	0.67
	27	2.08	0.19	0.36	0.53	0.70	0.87
	30	2.56	0.23	0.44	0.65	0.87	1.07
Ethyl acetate	35	2.97	0.26	0.50	0.75	0.99	1.24
	19	1.45	0.13	0.25	0.37	0.49	0.60
	26	2.45	0.21	0.42	0.62	0.81	1.02
	30	3.12	0.25	0.50	0.76	1.02	1.30
Butyl acetate	35	3.79	0.34	0.66	0.98	1.27	1.58
	19	0.27	0.024	0.05	0.07	0.09	0.11
	26	0.50	0.04	0.08	0.12	0.16	0.21
	30	0.76	0.06	0.12	0.18	0.25	0.31
Methyl ethyl ketone	35	0.97	0.07	0.15	0.24	0.32	0.40
	20	2.64	0.22	0.44	0.66	0.88	1.10
	26	3.27	0.31	0.59	0.87	1.11	1.36
	30	3.71	0.35	0.69	0.99	1.28	1.54
Benzene	35	4.09	0.38	0.76	1.08	1.39	1.70
	21	1.70	0.15	0.30	0.45	0.59	0.71
	25	2.75	0.24	0.49	0.72	0.93	1.14
	30	3.16	0.26	0.53	0.79	1.05	1.31
Trichloro-ethylene	35	3.57	0.29	0.59	0.89	1.20	1.48
	19	2.76	0.26	0.53	0.73	0.93	1.15
	25	3.92	0.29	0.60	0.89	1.17	1.47
	30	4.83	0.46	0.81	1.27	1.63	2.01
Acetone	35	5.91	0.57	0.97	1.55	2.00	2.46
	19	4.68	0.46	0.87	1.25	1.60	1.95
	26	5.35	0.50	1.01	1.47	1.86	2.23
	31	5.72	0.59	1.11	1.55	1.97	2.38
	35	6.17	0.64	1.18	1.64	2.12	2.57

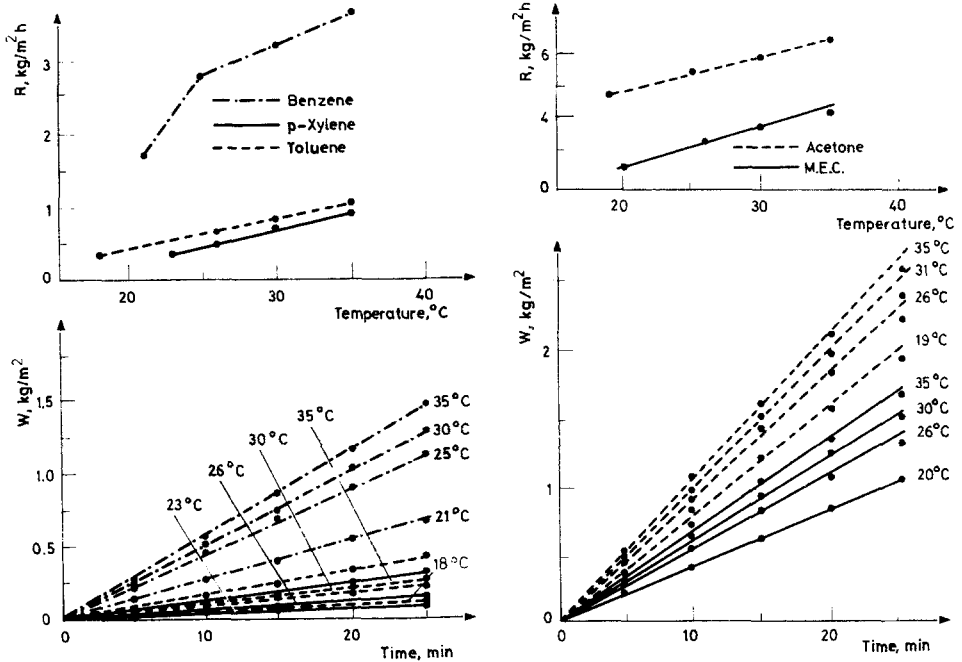


Fig. 2 Evaporation rates of benzene, toluene and p-xylene

Fig. 3 Influences of temperature and time on evaporation rates of acetone and methyl ethyl ketone

Chemically pure ethyl acetate displays a linear variation of the evaporation rate with temperature (Figs 4, 6), in contrast to technical ethyl acetate.

Benzene [4–7] and ethyl acetate [4, 5] form azeotropes with a great number of different solvents, which have boiling points in a wide temperature range. The boiling temperature gives an informative indication on the volatility of a solvent, its molar vaporization heat being a more adequate value [2].

The variation in the specific weight of the evaporated substance with time is also plotted in Figs 1–6.

The points lie on a straight line for the single solvents and solvent mixtures (Figs 1–5, 7, 8), showing the stability of the evaporation rate in this interval of time. The linear dependence no longer holds for the adhesive and film-forming materials, the slope decreasing in time. This is mainly due to the formation on the surface of a film which, although permeable to vapour, hampers the evaporation in time (Figs 9–11). The rates of formation and thickening of the film depend on the composition of the compound.

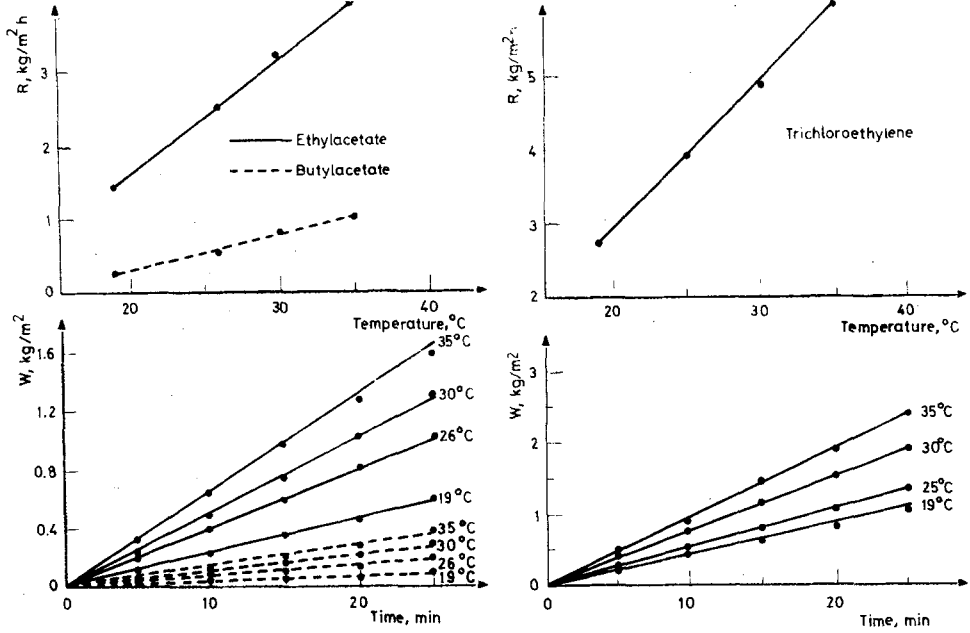


Fig. 4 Evaporation rates of ethyl and butyl acetates as functions of temperature and time

Fig. 5 Evaporation rates of trichloroethylene

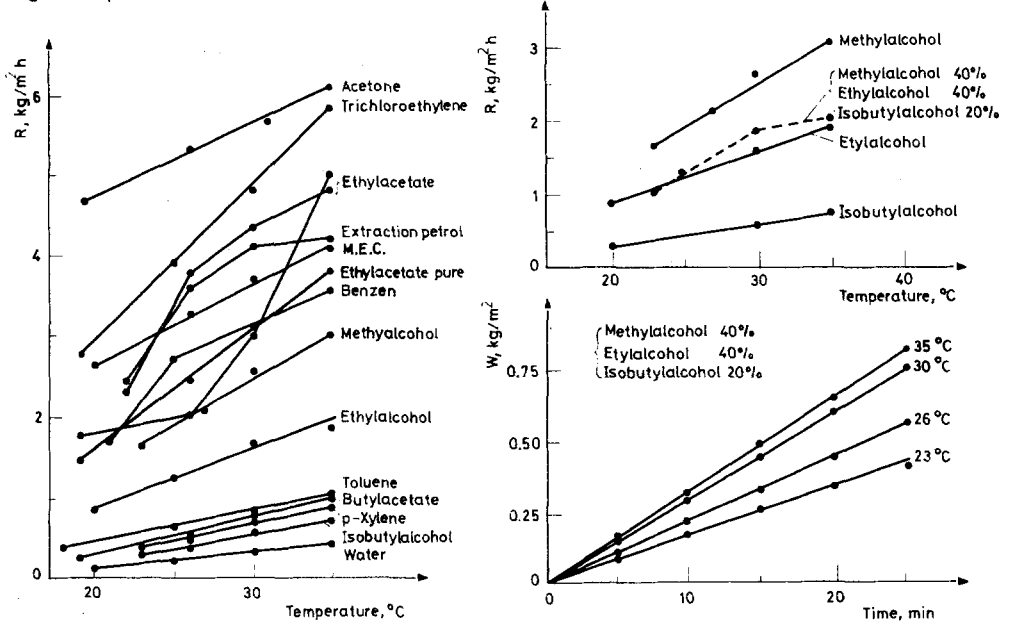


Fig. 6 Evaporation rates of the tested solvents as functions of temperature

Fig. 7 Evaporation rates of a mixture of three alcohols in comparison with the components of the mixture

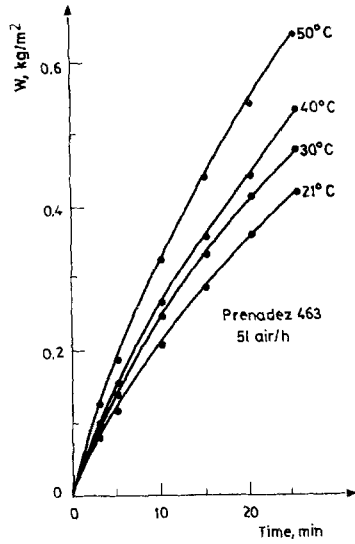
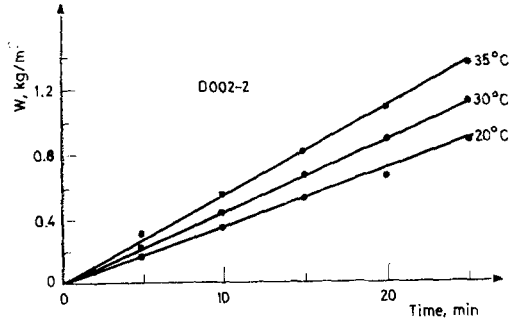
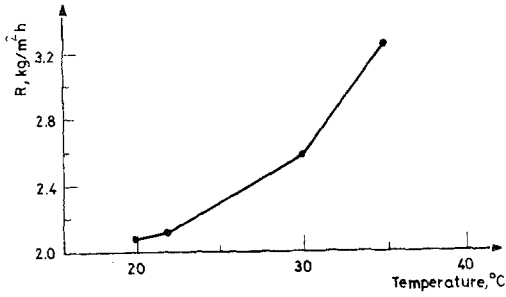


Fig. 8 Evaporation rates of the thinner for nitrocellulose lacquer

Fig. 9 Dependence of the evaporation rate of Prenadez 463 on time

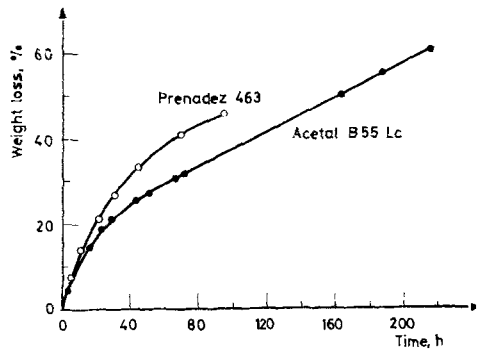
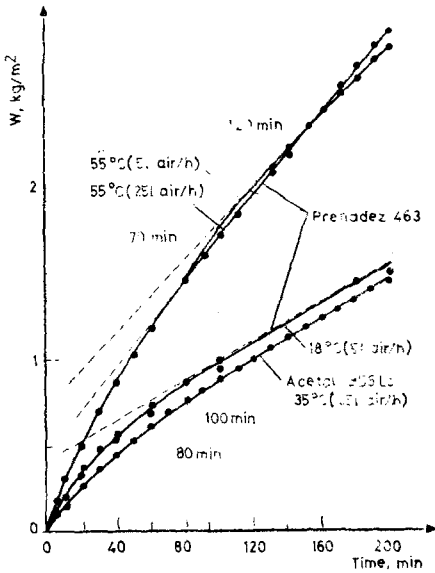


Fig. 10 Influences of temperature and oxygen quantity in air on film hardening

Fig. 11 Percentage loss in time of solvents from Prenadez 463 and Acetal B55 LC

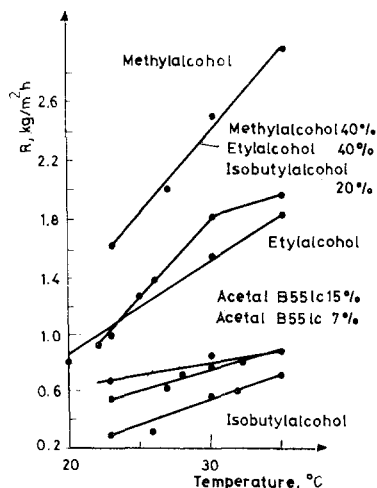


Fig. 12 Temperature dependence of evaporation rates of acetals B55 LC in comparison with the component solvents and their mixture

### Applications

Thermal analysis enables us to establish, under the same experimental conditions, the quantities of solvent vapour released from a solvent mixtures used as a thinner for nitrocellulose lacquers, whose evaporation rate increases rapidly with temperature (Fig. 8).

The evaporation rates were determined for an adhesive based on neoprene rubber (Prenadez 463, Fig. 9) and a lacquer for constructions based on polyvinyl butyral (B55 LC, Fig. 12), materials with very good adhesive and film-forming properties, which have been reported in the literature [8].

The decrease in the evaporation rate due to the formation and thickening in time of the film on the surface also enabled us to study the influences of temperature and oxygen from the air on the hardening rate. The material based on neoprene is more susceptible to oxidation, while that based on butyral hardens more slowly, the film preserving its permeability to vapour up to the quantitative exhaustion of the solvent (Figs 11, 12).

### Conclusions

The thermal analysis method used allows determination of the evaporation rates of toxic and inflammable solvents from materials containing them, with an average standard deviation of 4 per cent.

The gas-chromatographic determinations have demonstrated that during the release the same ratio is preserved between the solvents as in the initial composition.

## References

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**Zusammenfassung** – Aus den für verschiedene Temperaturen und Verdampfungsperioden erhaltenen TG-Kurven können die Thermoeffusiven Charakteristika von für adhäsive und filmbildende Materialien häufig verwendeten Lösungsmitteln ermittelt werden. Die Bestimmungen wurden in Luft bei einer Strömungsgeschwindigkeit von 5 l/h und im Temperaturbereich von 17–35° ausgeführt, wobei die Verdampfungsperiode bei jeder Temperatur 25 Minuten betrug. Untersucht wurden reine Lösungsmittel, Gemische mit Lösungsmittelverhältnissen, die denen in den Endprodukten entsprechen, sowie die Endprodukte selbst. Nach der vorgestellten Methode können die Verdampfungs geschwindigkeiten solcher Lösungsmittel mit einer Standardabweichung von durchschnittlich 4% bestimmt werden.

**Резюме** – ТГ-кривые, полученные при различных температурах и времени испарения, позволили установить термодиффузионные характеристики растворителей, часто используемых для связывающих и пленкообразующих веществ. Определения были проведены в атмосфере воздуха при объемной скорости потока 5 л/час с чистыми растворителями, с смесями растворителей в пропорциях, соответствующих тем, что в конечных продуктах, а также с конечными продуктами. Исследования проводились в температурном интервале 17–35°. При каждой температуре период испарения равнялся 25 минутам. Метод дает скорости испарения таких растворителей со средним стандартным отклонением в 4%.