TABLE II

PHYSICAL PROPERTIES OF REFERENCE LIQUIDS[a]

Property	Compound	Exp.	Lit.
Viscosity	Benzene	5.27, 5.26	5.26[b]
Surface	Water	70.56	70.85[4b]
tension (R)	Benzene	26.83	26.89[c]
B. p.	Nitrobenzene	210.9	210.9[d]
B. p.	Decalin	193.3	193.3[d]
B. p.	Ethylene glycol	197.4	197.5[d]
B. p.	*p*-Methoxy benzyl alcohol	259.1	258.8[d]
Interfacial	Chloroform	29.7 ± 0.5	32.8[e]
tension at 20°	Chlorobenzene	35.5 ± 0.2	37.4[e]
Interfacial tension at 35°	Carbon tetrachloride	43.2 ± 0.5	43.5[e]

[a] Units and experimental conditions are the same as in Table I unless otherwise specified. [b] "I. C. T.," Vol. V. [c] Hennaut-Roland and Lek, *Bull. soc. chim. Belg.*, **40**, 177 (1931); *C. A.* **25**, 5323 (1931). [d] "I. C. T.," Vol. I. [e] "I. C. T.," Vol. IV.

method of calculation of interfacial tension, including the use of a correction factor was that of Harkins.[4b] Table II indicates the comparison between our values and the literature values for several liquids. The agreement is as close at that reported by investigators using different experimental procedures.[6] The interfacial tension values for those liquids whose densities are only slightly different from that of water were either not determined, or if made, the precision was poor.

Summary

Physical constants for a large number of organic insect repellents were determined.

(6) Andreas, Hauser and Tucker, *J. Phys. Chem.*, **42**, 1001 (1938).

COLLEGE PARK, MARYLAND

RECEIVED SEPTEMBER 7, 1948

[CONTRIBUTION FROM THE CHEMICAL LABORATORIES OF THE POLYTECHNIC INSTITUTE OF BROOKLYN]

The Use of Radioactive Tracers in a Study of Activator Distribution in Infrared-Sensitive Phosphors[1,2]

BY R. W. MASON, C. F. HISKEY AND ROLAND WARD

The preparation and some of the properties of infrared-sensitive phosphors of the alkaline earth sulfides and selenides have been described by Urbach,[3] Smith,[4] Primak[5] and Stripp.[6] It has been found that reproducible samples are most conveniently prepared by the use of presumably non-reactive fluxes such as lithium fluoride or strontium chloride. The flux effects a complete recrystallization of the base material[5] and serves to introduce the activators during the recrystallization. Since the luminescent properties are quite sensitive to variation in the concentration of the activators, the relationship between these and the brightness of the phosphors under stimulation was investigated.[6] While the results were empirically satisfactory, the absolute activator concentration was not known since the activators at equilibrium conditions must be distributed between the base and the flux. The determination of distribution coefficients of rare earth ions at such low concentrations (0.02% or less) in alkaline earth compounds would be very difficult by the usual analytical procedures, but the availability of radioactive isotopes of europium, cerium and samarium offered a possible solution.

The main difficulty in the application of the tracer technique lay in the separation of the intimately mixed phases. A separation method based on the difference in specific gravities of the base and flux was only partially successful. A much more satisfactory procedure was devised which involved filtration at the fluxing temperature (about 1000°) using a platinum Munroe crucible. Since the method may find some application in studying solubility phenomena in molten salt systems, the procedure is described here in some detail.

Experimental

Materials.—Strontium sulfide and selenide were prepared as previously reported.[3,4] The fluxes, strontium chloride and lithium fluoride, contained only spectroscopic traces of the other alkaline earths and the alkali ions.

Procedure

(1) **Separation of Base and Flux by Filtration.**—The preparation of a Munroe crucible has been described by Snelling[7] and Swett.[8] Although the platinum mat is reasonably resistant to attack by the phosphor mixtures at 900 to 1100° in a nitrogen atmosphere, it eventually develops pinholes or cracks. These may be sealed by dipping in chloroplatinic acid and heating at 500° or by decomposing a layer of ammonium chloroplatinate on the mat and burnishing and compressing with a glass rod.

The mixtures containing strontium chloride separated readily by gravity filtration but those containing lithium fluoride required suction. For this purpose the apparatus shown in Fig. 1 was constructed. A smooth flange was forged on the Munroe crucible (1) and one end of the

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(1) The work described in this paper was carried out under Contract NObs 39045 between the Bureau of Ships and the Polytechnic Institute of Brooklyn.

(2) Abstracted from a thesis by R. W. Mason submitted to the Graduate School of the Polytechnic Institute of Brooklyn, 1948, in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

(3) F. Urbach, D. Pearlman and H. Hemmindinger, *J. Optical Soc. Am.*, **36**, 372 (1946).

(4) A. L. Smith, R. D. Rosenstein and R. Ward, THIS JOURNAL, **69**, 1725 (1947).

(5) W. Primak, R. K. Osterheld and R. Ward, *ibid.*, **69**, 1283 (1947).

(6) K. F. Stripp and R. Ward, *ibid.*, **70**, 401 (1948).

(7) W. O. Snelling, *ibid.*, **31**, 456 (1909).

(8) O. D. Swett, *ibid.*, **31**, 928 (1909).

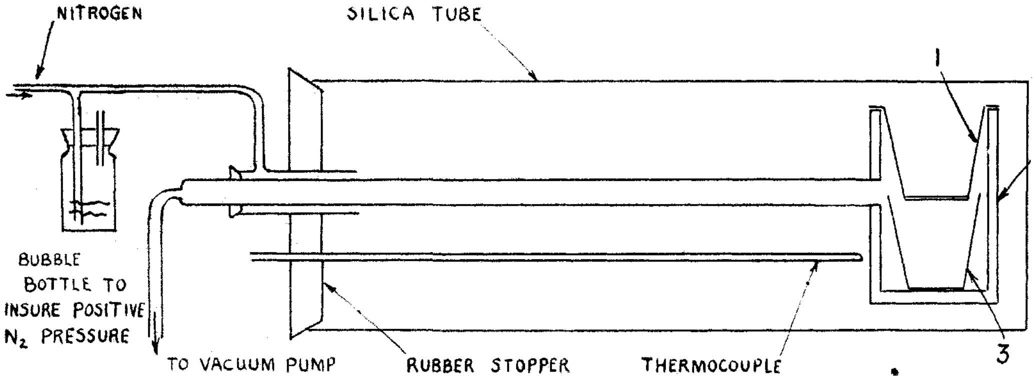

Fig. 1.—Apparatus for suction filtration.

silica tube (2) was faced by grinding with carborundum. The flange seated on this surface gave a sufficiently tight joint so that suction filtration could be rapidly completed. The filtrate was collected in the platinum receptacle (3). The platinum mat usually required repair after two or three suction filtrations. This may have been due to the applied pressure or to the more corrosive action of the lithium salts. It has been found that for some systems nickel is more resistant than platinum. Preliminary experiments showed that filtration could only be accomplished with mixtures containing a much higher proportion of flux than is used in the phosphor preparation (about 10%). For the systems strontium sulfide–strontium chloride and strontium selenide–strontium chloride, 60 to 85 weight per cent. of strontium chloride was found most suitable. Because of the high solubility of strontium sulfide in lithium fluoride, it was necessary with this system to use mixtures in the range of 40 to 60 per cent. flux.

The mixtures for distribution studies were prepared by adding to a weighed quantity of flux europium, cerium or samarium and an adequate amount of the radioisotope as solutions of their chlorides. The mixture was dried at 200° and mixed with the appropriate amount of base. To bring the system as close to equilibrium as possible before filtering, the mixture was pre-heated at the filtering temperature for one hour and cooled in an atmosphere of nitrogen. It was then transferred to the Munroe crucible and heated in a nitrogen atmosphere to the desired temperature. About one-half hour was sufficient for gravity filtration of the mixtures with strontium chloride. The lithium fluoride mixtures were heated for half an hour before filtering by suction which usually took less than a minute.

After cooling under nitrogen, the filtrate and residue were powdered and samples of each reserved for counting and for chemical analysis. The solubilities of the base materials in the molten fluxes are given by analyses of the filtrates.

(2) **Analyses of the Fractions.**—For the strontium chloride–strontium sulfide mixtures, the determination of the sulfide sulfur was made by taking a portion of the fraction, adding it to a standard iodine solution, and after acidification titrating the excess iodine with thiosulfate solution. The strontium chloride was found by difference and the total strontium by conversion to the sulfate. The same general method was followed with the strontium chloride–strontium selenide mixtures except that selenium was determined by oxidation with bromine in acid solution and precipitation as selenium by hydrazonium ion. The lithium fluoride–strontium sulfide mixtures were taken to dryness with sulfuric acid and weighed. The lithium sulfate was dissolved in water and alcohol was added to give a 50% solution. The strontium sulfate was filtered and weighed and the lithium determined by difference. These procedures were checked by more complete standard analyses and were found to be sufficiently precise for this work. The precision was about ±0.2% for the strontium chloride systems and about ±0.5% for the lithium fluoride–strontium sulfide mixtures.

Some of the results are given in Tables I, II and III. From the analyses of the filtrates it can be seen that the solubilities of the strontium sulfide and strontium selenide in the strontium chloride are quite low compared with the solubility of

Table I

Filtration of Strontium Chloride–Strontium Sulfide Mixtures

Sample	Temp., in °C.	Weight % SrS in Unfiltered mixtures	Weight % SrS in Filtrate	Weight % SrS in Residue
1	900	25	2.6	36.3
2	900	25	2.5	33.1
3	900	25	2.7	34.0
4	900	25	2.5	35.4
5	1050	20	3.7	44.5
6	1050	20	3.7	43.5
7	1050	40	4.1	51.4
8	1050	40	3.7	48.0

Table II

Filtration of Strontium Chloride–Strontium Selenide Mixtures

Sample	Temp., in °C.	Weight % SrSe in Unfiltered mixtures	Weight % SrSe in Filtrate	Weight % SrSe in Residue
9	920	40.0	5.0	49.2
10	920	40.0	4.7	49.7
11	920	40.0	4.3	49.8
12	1050	40.0	6.1	45.7
13	1050	25.0	5.8	43.5
14	1050	25.0	7.1	47.4
15	1050	40.0	7.1	50.0

Table III

Filtration of Lithium Fluoride–Strontium Sulfide Mixtures: Fifteen-gram Samples Containing Sixty Weight Per Cent. SrS

Sample	Temp., in °C.	Weight % SrS in Filtrate	Weight % SrS in Residue
16	940	35.8	82.0
17	1000	37.3	82.0
18	1000	38.3	79.0
19	1000	37.6	80.0
20	1100	45.2	90.0

strontium sulfide in lithium fluoride. The solubilities of the sulfide in the strontium chloride and in the lithium fluoride show an average deviation of about 4%, but for the selenide the average deviation is about 9%. An appreciable increase in solubility with rising temperature is apparent in each of the systems.

(3) **Activator Distribution.**—Samples of the filtrates and residues were prepared for counting by a slight modification of the procedure described by Kamen.[9] In most instances the sample was ground, a 50- or 100-mg. portion suspended in symmetrical tetrachlorethane and filtered through a circular paper in a gooch crucible. The paper, thus covered with an even layer of solid, was transferred to a copper plate, dried and fixed in position with scotch tape. The sample was then bent to fit around the counter tube. The reproducibility was quite satisfactory. It was found that no correction was required for self-absorption. A few samples were prepared by dissolving the sample and evaporating an aliquot on absorbent paper. The precision of the counting including the preparation of samples was about ± 5%.

In Table IV are given the characteristics of the radioactive samples. They were prepared at the Oak Ridge laboratories by pile irradiation of pure compounds of the elements. The compounds used were europium oxide (furnished by the late Herbert N. McCoy), cerous nitrate and samarium oxide (prepared in Luigi Rolla's laboratory). In counting the cerium samples the beta-radiation was eliminated by means of aluminum foil so that only the gamma-radiation from the cerium isotopes was counted. A standard sample was counted periodically during the course of the measurements. The data, plotted in Fig. 2 against the theoretical decay curve for a twenty-eight day half-life, indicate the possibility that a longer lived contaminant was present, but in negligible amounts. The decay of the samarium sample is shown in Fig. 3 to be close to the theoretical forty-seven hour half-life curve which indicates that the contribution of europium 155 to the total count is unimportant.

TABLE IV

CHARACTERISTICS OF RADIOACTIVE ACTIVATOR SAMPLES[10]

Irradiated sample	Radioisotopes present	Half-life	Radiation (MEV)	
			Beta	Gamma
Europium	Eu 152 ,	9.2h	1.61	
	154	5.8y	0.34, 0.82	1.4 et al.
Cerium	Ce 141	28d	0.55	0.21
	143 (Parent of Pr 143)	33h	1.35	0.50
	Pr 143	13.8d	0.95	None
Samarium	Sm 153	47h	0.73	0.1, 0.57
	155 (Parent of Eu 155)	25m	1.9	0.3
	Eu 155	2y	0.3	0.084

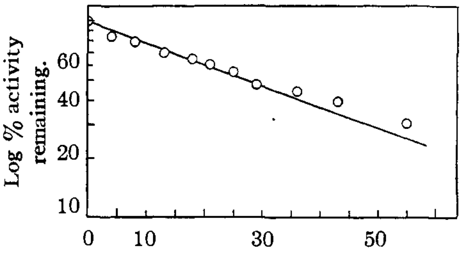

Fig. 2.—The line represents the theoretical decay for twenty-eight day half-life.

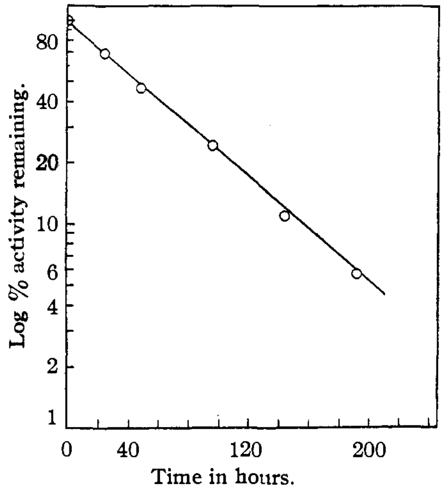

Fig. 3.—The line represents the theoretical decay for forty-seven hour half-life.

The quantities of irradiated europium, cerium and samarium which gave convenient counts in the distribution studies were approximately 10, 200 and 3 parts per million of the base material respectively. Additional quantities of the non-radioactive activators were added so that the distribution could be determined at different activator concentrations.

(a) **Distribution of Europium.**—The data obtained for the calculation of the distribution coefficient of europium in the different systems are listed in Tables V, VI and VII. The numbers in brackets refer to the sample numbers in Tables I, II and III. A four- to eightfold variation in activator concentration was used.

In this report the distribution coefficient is defined as the ratio of the mole fractions of europium in the solid phase and in the liquid phase, *i.e.*

$$K = \left[\frac{n_{\mathrm{Eu}}(\mathrm{s})}{(n_{\mathrm{Eu}}(\mathrm{s}) + n_{\mathrm{B}})}\right]_{\text{solid phase}} \times \left[\frac{n_{\mathrm{Eu}}(\mathrm{l}) + n_{\mathrm{B}} + n_{\mathrm{F}}}{n_{\mathrm{Eu}}(\mathrm{l})}\right]_{\text{liquid phase}}$$

where $n_{\mathrm{Eu}}(\mathrm{s})$ and $n_{\mathrm{Eu}}(\mathrm{l})$ are the number of moles of europium in the solid and liquid phases, respectively, and n_{B} and n_{F} are the number of moles of base and flux, respectively. This involves the reasonable assumption that none of

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(9) M. D. Kamen, "Radioactive Tracers in Biology," Academic Press, New York, N. Y., 1947, p. 152.

(10) Data taken from Radioisotopes Catalog and Price List No. 2, U. S. Atomic Energy Commission, Oak Ridge, Tennessee, revised September, 1947.

the flux dissolves in the base but that only base dissolves in flux. Hence at equilibrium we have the pure base in contact with a saturated solution of the base in the fluid flux. It can be seen that this does not quite represent the distribution in the actual phosphor which consists of a mixture of crystals of pure flux and base. Where the solubility of the base in the flux is low, however, it may be assumed that the distribution coefficient in the solids is not very different.

The distribution coefficient may be calculated from the formula[11]

$$K = \frac{(a + b)[x - y(c/a)]}{y[d - b(c/a)]}$$

where a = moles flux per 100 g. of filtrate
b = moles base per 100 g. of filtrate
c = moles flux per 100 g. of residue
d = moles base per 100 g. of residue
x = counts per standard weight of residue
y = counts per standard weight of filtrate

The values listed for K in Tables V to VII indicate that equilibrium had been fairly well established. With the strontium chloride flux, the activator distribution is not appreciably affected by change of temperature, variation of flux to base ratio or by change of total activator concentration. The concentration of the activator in the liquid phase is somewhat more than one third of the concentration in the solid phase. In the actual phosphor mixture, which contains only about 10% flux, however, about 97% of the added activator resides in the base material.

TABLE V

DISTRIBUTION OF EUROPIUM BETWEEN STRONTIUM CHLORIDE AND STRONTIUM SULFIDE

	Parts Eu per million parts SrS in unfiltered mixture	Counts per minute Filtrate	Counts per minute Residue	K
(1)	200	1069	2045	2.8
(2)	100	1158	1970	2.5
(3)	400	1233	2219	2.7
(4)	800	1084	1977	2.6
(5)	100	98.4	230	3.2
(6)	400	127.0	231	2.3
(7)	100	68.0	157	2.8
(8)	400	66.8	137	2.5

TABLE VI

DISTRIBUTION OF EUROPIUM BETWEEN STRONTIUM CHLORIDE AND STRONTIUM SELENIDE

	Parts Eu per million parts SrSe in unfiltered mixture	Counts per minute Filtrate	Counts per minute Residue	K
(9)	50	761	1217	2.4
(10)	100	744	1142	2.3
(11)	400	790	1206	2.2
(12)	800	677	1085	2.6
(13)	100	978	1503	2.5
(14)	400	768	1206	2.5
(15)	400	937	1345	2.1

TABLE VII

DISTRIBUTION OF EUROPIUM BETWEEN LITHIUM FLUORIDE AND STRONTIUM SULFIDE

	Parts Eu per million parts SrS in unfiltered mixture	Counts per minute Filtrate	Counts per minute Residue	K
(16)	150	207	1647	35.3
(17)	500	320	1623	21.9
(18)	1000	370	1690	20.7
(19)	150	314	1734	24.4
(20)	150	611	2272	13.2

In the case of the strontium sulfide–lithium fluoride system, on the other hand, the values of the distribution coefficient (Table VII) are much higher and there is a pronounced decrease with rising temperature. At 1000° the distribution coefficient for this system is more than ten times that for the strontium chloride system. The solubility of the sulfide in the lithium fluoride is also about ten times that in strontium chloride. This means that when the mixture is cooled from 1000° the portion of the sulfide which crystallizes out from solution will have a much lower activator concentration than that which was undissolved at 1000°. With the usual phosphor mixture containing about 10% flux, only 3 to 4% of the sulfide would have dissolved at 1000° and this portion on solidification of the mixture would have an activator concentration about one seventh of that of the main bulk of the sulfide if we assume that no activator remains with the lithium fluoride solid phase. The assumption that the activator concentration in the solid lithium fluoride is essentially zero was justified by the separation of the solid phases into several fractions containing different proportions of flux and base. This was accomplished by taking separate samples of the filtrate and residue from a filtration carried out at 1100° and grinding them under tetrabromoethane to a paste containing particles about 10–20 microns in size. The paste was suspended in a mixture of tetrachloro- and tetrabromoethanes and centrifuged. This was repeated with liquids of progressively higher densities containing more tetrabromoethane so that a series of floating fractions with increasingly higher concentrations of the heavier component were obtained. The fractions were analyzed and counted and the results extra-

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(11) This expression is derived from the distribution coefficient defined above in the following manner: (a) It is apparent that the number of moles of activator, n_{Eu}, is trivial in comparison to the number of moles of the base or the flux. Thus

$$(K = [n_{\mathrm{Eu}}(\mathrm{s})/n_{\mathrm{B}}]_{\mathrm{solid}}\,[n_{\mathrm{B}} + n_{\mathrm{F}})/n_{\mathrm{Eu}}(1)]_{\mathrm{liquid}}$$

(b) The determination of $[n_{\mathrm{Eu}}(\mathrm{s})/n_{\mathrm{B}}]_{\mathrm{solid}}$ would, under ordinary filtration techniques, merely involve weighing the undissolved base and making a count to estimate the europium present. Unfortunately, this cannot be done in this case because along with the undissolved base there is always a significant quantity of filtrate retained on the filter, and this filtrate contains dissolved base as well as radioactive Eu. It becomes necessary, therefore, for us to subtract this dissolved base from the total base concentration in the residue and the radioactivity count from the total count observed for the residue. In terms of the analytical quantities a, b, c, d, x and y defined previously, the expression for n_{B} is thus $[d - b(c/a)]$ and for $n_{\mathrm{Eu}}(\mathrm{s})$ is $k[x - y(c/a)]$ where k is a proportionality constant. (c) The filtrate is merely analyzed for its count and the number of moles of flux and base in it. The term $[n_{\mathrm{Eu}}(\mathrm{l})/(n_{\mathrm{F}} + n_{\mathrm{B}})]_{\mathrm{liquid}}$ is thus equal to $ky/(a + b)$.

polated to the count corresponding to one of the pure components.

The results given in Table VIII are plotted in Fig. 4 which shows two curves, one connecting the points representing the separated fractions of the filtrate and one connecting the points representing the fractions of the residue. The two curves are due to the different concentrations of europium in the residue and filtrate. Both curves extrapolate to zero concentration of europium in the lithium fluoride. The deviation from linearity is probably due to inhomogeneous distribution of the europium.

TABLE VIII

LITHIUM FLUORIDE–STRONTIUM SULFIDE–EUROPIUM SINK-FLOAT SEPARATIONS

	Sp. gr. of fraction	% of total sample	Wt. % SrS in fraction	Counts per min. per 0.05 g. of fraction
Residue from filtration	<2.75	0	..	..
(69.7% SrS	2.75–2.85	3.5	27.8	232
1364 counts per min.	2.85–2.95	18.2	27.7	482
per 0.05 g. sample)	>2.95	78.3	89.0	1835
Filtrate	<2.77	10.7	12.9	69
(39.7% SrS	2.77–2.88	42.2	25.9	193
366 counts per min.	2.88–2.93	17.8	25.2	195
per 0.05 g. sample)	>2.93	29.3	81.4	814

From the results it would appear that in these phosphor systems the assumption can be made that essentially all of the europium added to the mixture finds its way into the lattice of the base material. With the lithium fluoride flux, the phosphor will be slightly inhomogeneous.

(b) **Distribution of Cerium and Samarium.**—The experimental results for the distribution of cerium and samarium in the same systems are given in Tables IX and X. Again it can be seen that the distribution coefficients are much larger for the strontium sulfide–lithium fluoride system than for the systems with the strontium chloride flux. The distribution coefficients for cerium in the strontium sulfide systems are lower than those for samarium.

TABLE IX

DISTRIBUTION OF CERIUM AND SAMARIUM. COMPOSITION OF FILTRATES AND RESIDUES

Sample	Temp., in °C.	Weight % base in Unfiltered mixtures	Filtrate	Residue
1	1000	25.0 SrS	3.8 SrS	37.2 SrS
2	1000	30.0	3.8	45.0
3	1000	40.0	3.6	47.7
4	1000	25.0	3.9	42.1
5	1000	40.0	4.7	48.7
6	1000	25.0 SrSe	6.5 SrSe	44.4 SrSe
7	1000	30.0	6.0	45.8
8	1000	40.0	5.5	52.8
9	1000	25.0	7.0	43.6
10	1000	40.0	7.5	53.1
11	920	60.0 SrS	28.4 SrS	73.6 SrS
12	1080	60.0	39.4	79.2
13	900	60.0	25.0	74.8
14	1060	60.0	38.3	77.6

Experiments 1– 5: SrS–$SrCl_2$
6–10: SrSe–$SrCl_2$
11–14: SrS–LiF

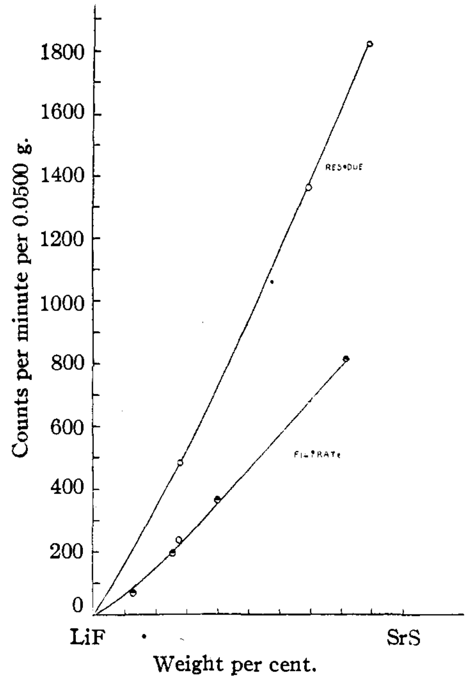

Fig. 4.—Distribution of europium in the LiF–SrS system by sink-float separation of phases.

TABLE X

DISTRIBUTION OF CERIUM AND SAMARIUM IN THE VARIOUS SYSTEMS

Sample[12]	Parts activator per million parts base in unfiltered mixture	Counts per minute Filtrate	Residue	K
1	800 Ce	128	168	1.4
2	500 Ce	122	149	1.2
3	223 Ce	91	110	1.1
4	200 Sm	540	1642	4.7
5	25 Sm	543	1186	2.7
6	800 Ce	123	216	3.0
7	500 Ce	104	142	2.0
8	223 Ce	111	122	1.3
9	200 Sm	829	1333	2.7
10	25 Sm	1555	968	0.24
11	200 Ce	66	200	15
12	200 Ce	67	239	16
13	100 Sm	219	1845	45
14	100 Sm	127	1491	58

There is, however, a marked difference in the distribution coefficients for these activators with changing composition of the original mixtures.

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(12) Samples correspond to those in Table IX.

In these experiments, unfortunately, both the percentage base and the activator concentration were changed so that it is not clear from the data whether the distribution coefficients decrease with decreasing activator concentration or decrease with increasing percentage of base. It seems reasonably certain from additional experiments that the latter alternative represents the situation. The solubility of strontium oxide in strontium chloride has been found to be quite high and its presence in the flux causes a pronounced decrease in the distribution coefficient of the activators. The effect with the different activators increases in the order europium–cerium–samarium. The strontium sulfide and selenide contain small amounts of oxide which dissolve in the flux so that increasing proportions of the base material in the original mixtures give rise to increasing concentrations of oxide in the flux. This leads to smaller distribution coefficients. The results of a systematic study of this phenomenon will be presented in a later publication.

Acknowledgment.—The authors wish to thank Dr. John R. Dunning and various members of the Radiochemical Laboratories at Columbia University for their friendly coöperation. We are particularly indebted to Mrs. Adele Weil for advice on tracer techniques.

Summary

A technique for filtering fused salt solutions at 1000° has been described.

The distribution of small quantities of europium, cerium and samarium between phosphor base materials (strontium sulfide and strontium selenide) and saturated solutions of the bases in the fluxes (strontium chloride and lithium fluoride) has been studied in the neighborhood of 1000° with the aid of radioactive tracers.

BROOKLYN 2, N. Y. RECEIVED JULY 3, 1948

[CONTRIBUTION FROM THE CHEMISTRY DEPARTMENT, UNIVERSITY OF PITTSBURGH, AND MELLON INSTITUTE]

Some Physical Properties of Isoquinoline

BY HENRY FREISER[1] AND WILLIAM L. GLOWACKI[2]

This paper is the second in a series[3] describing the work of a program for the determination of physical constants of coal tar bases, sponsored by the Koppers Company.

Purification of Isoquinoline.—Seventeen gallons of commercial isoquinoline (Koppers Company 2° isoquinoline having a freezing point of 23.8°) was subjected to eight successive fractional crystallizations until the final material (about 2 kg.) had a freezing point of 26.6°. Further purification was effected by a careful fractional distillation at atmospheric pressure through the rectifying column previously used.[3] Fifty-eight fractions of 30 ml. each were collected at the rate of 30 ml./hr., and after the first three fractions boiled within 0.1°. It is interesting to note that the distillation separated a high-melting impurity more volatile than isoquinoline. This material was identified as naphthalene and is believed responsible for the difficulty encountered in the purification by crystallization. The distillation cuts were further screened by measuring freezing points. For all of the physical properties determined, those central cuts whose freezing points differed by less than 0.05° were combined to give a material having a freezing point of 26.41 ± 0.02° and an estimated purity of at least 99.5 mole per cent.

Determination of Properties.—The properties reported here were determined using the same apparatus and procedures previously described.[3]

Freezing Point.—The freezing point of pure isoquinoline is estimated at 26.48 ± 0.1°. Quinoline was found to depress the freezing point of isoquinoline by 0.5 ± 0.1° per mole per cent. If the only impurity in the isoquinoline used were quinoline, the material might be as high as 99.8 mole per cent. pure.

Density and Expansion Coefficient.—The density values at every 10° from 30 to 80° were found to be 1.09101, 1.08309, 1.07519, 1.06731, 1.05945, and 1.05143 g./ml. with an average reproducibility of 0.00004 g./ml. The values of the expansion coefficient in this temperature range varied from 0.000722 at 30° to 0.000776 at 80°. Values of the density at high temperatures were determined as follows: 1.03540 at 100°, 1.01547 at 125°, 0.99498 at 150°, 0.97421 at 175°, and 0.95300 g./ml. at 200°, with an average reproducibility of 0.00006 g. per ml. The corresponding expansion coefficient values varied from 0.000765 to 0.000899.

Viscosity.—The results are presented in Table I.

TABLE I

VISCOSITY OF ISOQUINOLINE

Temp., °C.	Viscosity, cp.	Temp., °C.	Viscosity, cp.
30	3.2528	100	1.0230
40	2.6034	125	0.7787
50	2.1323	150	.6217
60	1.7872	175	.5067
70	1.5269	200	.4223
80	1.3223		

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