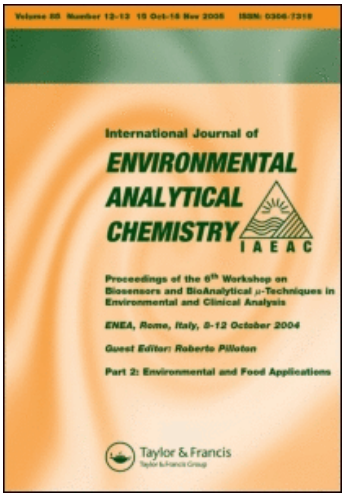

International Journal of Environmental Analytical Chemistry

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

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To cite this Article Krause, W. J. and Albrecht, H.(1977) 'Examinations of Filter Materials and Adhesives by Activation Analysis', International Journal of Environmental Analytical Chemistry, 5: 1, 35 — 46
To link to this Article: DOI: 10.1080/03067317708071128
URL: http://dx.doi.org/10.1080/03067317708071128

Intern. J. Environ. Anal. Chem., 1977, Vol. 5, pp. 35–46

Printed in Great Britain

Examinations of Filter Materials and Adhesives by Activation Analysis

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(*Received June 15, 1976*)

For activation analysis of filter precipitates by simply irradiating the filters together with the precipitates in a reactor the composition of the filter material must be known. However, several filter materials become brittle and break into pieces by neutron irradiation. To maintain a defined sample geometry for the gamma spectrometry it is appropriate to fix the filters in an envelope of polyethylene foil by means of an adhesive.

To select materials with minimum background activity after activation, membrane filters, glass fiber filters and adhesives of different manufacturers were examined for composition. After irradiation times of 1 hour and a flux of $8 \cdot 10^{13}$ n/cm^2 · sec up to 30 elements per sample were quantitatively determinable.

For all examined blank filters and adhesives the main components consist of Na, Cl, K, Fe, Zn and Br. Glass fiber filters show nearly a 100-fold higher Na content and also a considerable Ba content.

Membrane filters of polyamide and teflon and the Dekalin adhesive were found to have the best properties.

KEY WORDS: Instrumental neutron activation analysis (INAA), Filter material composition, Membrane filters, Adhesives.

INTRODUCTION

A simple method for analytical activation analysis of inactive precipitates on membrane or glass fiber filters consists of irradiating the precipitates together with the filter materials in a reactor and analyzing them by gamma spectrometry. Applying this method the question is to know the composition of the filter materials and the resulting background activity. The chemical analysis of the filters and especially the concentration of trace elements generally could not be obtained from the manufacturers. Therefore it was necessary to carry out corresponding investigations.

Similar examinations, known until now, on filter materials were performed mainly for determination of the environmental burden due to emitted dust and aerosols or for trace analysis in criminalistics. In these previous studies, filter materials of cellulose and their derivatives from American, English and German manufacturers, of polystyrene, glass fiber filters, as well as paraffin, cotton, collodion and several glasses were examined (Table I). For many elements detection limits in the lower nanogram range, sometimes even lower, have been reached and up to 25 different elements could be detected in the mentioned filter types.

In the present case, precipitates of aerosols of different fission product elements, as well as UO_2, steel and zircaloy had to be analyzed within the scope of experimental core melting investigations.[1] Due to the special experimental conditions the filters had to meet special requirements which were not satisfied by the filters examined earlier.

These special requirements were:

temperature resistance to at least 120°C at a relative humidity of >90%,
high retention factor (>99%) for the aerosols of corium components,
no comparable concentration of the elements to be analyzed in the precipitate and
no other elements activatable to a signifcant extent.

According to the expected aerosol particle size, filters with a pore size between 0.2 and 10 μm were used. The flow rate of air through the filters at a pressure of 500 mm of water was between 3 and 100 l/min. Filters that meet these conditions are offered in the Federal Republic of Germany only by the firms Sartorius-Membranfilter, Millipore-Filter and Schleicher + Schüll. Based on the mentioned requirements tests were conducted on membrane filters of cellulose or their derivatives, teflon and polyamide, as well as glass fiber filters (Table II). Before irradiation, the loaded filters of 50 mm diameter were sealed into 6 × 6 cm polyethylene envelopes to protect the precipitate against abrasion loss or contamination.

EXPERIMENTAL

The use of adhesives to fix filters in its envelope

At the beginning of the examinations we observed that the reactor neutron irradiation deteriorates the structure of the filters containing cellulose. They became brittle and broke into small pieces, some of which had the size of dust particles. On the contrary, polyamide and teflon filters were resistant to irradiation.

Therefore, to assure a reproducible measuring geometry for gamma spectrometric analysis these filters were fixed to the protective polyethylene

TABLE I

Filter materials examined by activation analysis (1970–1974)

Filter material	Filter type	Manufacturer	References
Celluose	Type C	C. D. Dexter & Sons (USA)	2
Cellulose	White band 589/2	SS (D)	3
Cellulose	Whatman 2, 40, 42	RA (USA)	4
Cellulose	Whatman 44 and 50	RA (USA)	4
Cellulose	Whatman 1 and 541	RA (USA)	8
Cellulose	Whatman 41	Balston Ltd. (GB)	2, 3, 5, 6
Cellulose	Binzer Qual. 4	Binzer (D)	3
Cellulose acetate	Cellotate EH	MiF (USA)	2, 4, 7
Cellulose acetate	MCB 20 and 25	unknown	8
Cellulose acetate	EK batch A-2X	unknown	8
Cellulose acetate	Metricel GA-6	GI (USA)	2
Cellulose nitrate	MF SM 113	SM (D)	3
Cellulose ester	MF Millipore WS	MiF (USA)	4
Cellulose ester	MF Millipore HA, AA	MiF (USA)	2
Polystyrene	Microsorban DS, DMS	Delbag (D)	2, 7
Polystyrene	Mikrosorban SM 159	SM (D)	3
Glass fiber	SS Shark Skin	SS (D)	4
Glass	NBS 50 (SRM G12)	NBS (USA)	6

Abbreviations: SS: Schleicher & Schüll
RA: Reeve Angel & Co.
MF: membrane filter
MiF: Millipore Filter Ltd.
GI: Gelman Instruments
NBS: National Bureau of Standards
SM: Sartorius Membranfilter

TABLE II

Filter materials examined by activation analysis in recent investigation

Filter material	Filter type	Manufacturer
Regenerated cellulose	MF SM 11606/07	SM
Regenerated cellulose	Selectron RCT-02	SS
Cellulose acetate	MF SM 11106/07	SM
Cellulose acetate	Selectron OE-66	SS
Cellulose nitrate	MF SM 13303/11306	SM
Cellulose nitrate	Selectron AE-91	SS
Cellulose nitrate	Selectron BA-83	SS
Cellulose ester	MF Millipore SC, SM, AA and HA	Mif
Polyamide	MF SM 11905	SM
Teflon	Mitex LS	MiF
Teflon with polyethylene gauze	Fluoropore FH	MiF
Glass fiber	SM 13400	SM

Abbreviations: see Table I.

foil. Since during the neutron irradiation of the filters the adhesives were activated too, they had to satisfy the same requirements as the blank filters (see above). Beyond that, the following conditions had to be fulfilled for the adhesives:

no chemical decomposition nor reaction with the filter material or with the polyethylene foil,
to be free of chlorine, so that no radioactive Cl-38 (undesired in the reactor irradiation channel) can escape,
to be resistant up to a temperature of at least 120°C which is caused by the irradiation, and
not to form gaseous products until this temperature.

From the manufacturers no information concerning the chemical composition of the adhesives (Table III) was available, especially not about chlorine content.

TABLE III

Adhesives examined by activation analysis

Trade name	Manufacturr
Uhu-Alleskleber (all adhesive)	Lingner + Fischer/Bühl
Uhu-Papier-fit (paper adhesive)	Lingner + Fischer/Bühl
Uhu-hart	Lingner + Fischer/Bühl
Pelikan-Gummi (gum)	Günter Wagner, Pelikan-Werke/Hannover
Pelikanol-Weissleim (glue)	Günter Wagner, Pelikan-Werke/Hannover
Haftkleber 1188 (adhesive)	Dekalin, Deutsche Klebstoffwerke/Hanau
Ipacollan B 50 CS	Isar-Rakoll-Chemie/München
Snap-Fix Photo Gum	A. Faber/Neu Isenburg
Pritt-Stift (adhesive pin)	Henkel Cie./Düsseldorf
Pattex	Henkel Cie./Düsseldorf
Elefantenkleber (gum)	Helios-Chemie/Hannover

Sample preparation for neutron irradiation

The following sample types were examined: blank envelopes of polyethylene foil (about 70 cm^2), blank filters, and adhesives that were put separately in polyethylene foil envelopes. Two or three of these samples were sealed together into a larger polyethylene envelope and loaded into a capsule for irradiation in a pneumatic tube. To avoid that the densely packed foils stick together during irradiation due to heating up to nearly 120°C, 0.01 mm aluminum foils were placed between the polyethylene foils.

Except for the Selectron filters, 3–5 identical samples of each filter material and 8 blank samples of the polyethylene foil were examined. The adhesives were distributed as thinly and uniformly as possible (200–300 mg average). The samples were irradiated in the pneumatic installation of the Karlsruhe FR-2 reactor at a neutron flux of $8 \cdot 10^{13} n/cm^2 \cdot sec$. The irradiation time was usually one hour, but for the glass fiber filters it was 15 minutes.

After a cooling time of at least one hour for decay of radionuclides with half lives < 15 minutes, the samples were taken from the pneumatic capsule and sealed into larger polyethylene foil envelopes free from contamination. In this form they were analyzed 3 times by gamma spectrometry after approximately 3,100 and 600 hours of decay time.

System for gamma spectrometric measurements

The samples were measured with a Ge(Li)-semiconductor detector which had a volume of 84.5 cm^3, an efficiency of 13 %, a resolution of about 2.0 keV and a peak-to-Compton ratio of 42:1 for the upper Co-60 peak. Detector and preamplifier were situated in a box shielded with 5 cm of lead and coated inside with Cd, Cu and plexiglass. In addition, this system included a 4096 channel analyzer, a PDP-11 computer with 16 K memory and several devices for data input and output (Figure 1).

Using the irradiation data, the measured spectra, decay times and the characteristic nuclide data (half lives,[9] (n,γ) and (n,p) cross-sections,[9,12] gamma ray intensities[10–12]) the activities and masses of the identified radionuclides as well as the masses of the elements originally present were determined.

RESULTS AND DISCUSSION

Polyethylene foil and filter materials

After one hour of irradiation time, the membrane filter without the irradiated polyethylene foil had an initial activity between 60 and 200 μCi. After 15 minutes of irradiation the glass fiber filters showed an initial activity of about 4 mCi consisting mostly of Na-24 activity (about 95 %).

For all filters about 30 elements above atomic number 10 were found (Table IV). The total concentration of these elements in membrane filters was between 10 and 130 μg/filter. The main components were Na, Cl, K, Fe, and Zn. The first spectra taken after the irradiation contained the 1294 keV peak of Ar-41, resulting from the air enclosed in the envelopes.

Glass fiber filters showed nearly a 1000-fold higher Na and K content, about a 10,000-fold higher Zn content and moreover a considerable amount of Ba.

TABLE IV

Elemental components and impurities in various filter materials (in microgram/filter, resp./70 cm^2 of foil)

	enerated Cellulose			Cellulose acetate			Cellulose nitrate				Cellulose ester Millipore SC, SM, AA, HA		Polyamide SM 11905		Teflon Millipore Mitex LS		Teflon w Polyethy Fluorop FHLF	
	1606/07	S+S RCT-02		SM 11106/07	S+S OE-66		SM 13303/11306	S+S AE-91	S+S BA-83									
	D	43		16	D	11	37	C	30	24	31	C	7.8	D	9.7	E	9.8	
	D	n.m.		8.7	D	n.m.	11	D	n.m.	n.m.	25	D	14	E	<0.1	I	0.26	
	D	n.m.		8.9	D	n.m.	9.4	D	n.m.	n.m.	6.0	E	10	D	<0.1	I	<0.1	
	C	6.0		<0.1	I	0.4	2.3	F	n.m.	n.m.	1.8	E	0.33	G	1.4	F	3.3	
0018	E	0.00008		0.00021	F	0.00012	0.00001	G	n.m.	0.00009	0.000003	I	0.000053	F	0.00013	D	0.00011	
088	E	0.0003		0.014	E	0.010	0.0033	F	0.00025	0.014	0.14	C	0.0015	G	0.079	C	0.020	
076	F	n.m.		0.023	E	n.m.	0.037	D	n.m.	n.m.	0.040	D	0.010	E	0.038	D	0.024	
	D	0.94		2.6	D	0.42	2.0	D	1.3	3.3	0.55	E	0.41	F	0.76	D	0.58	
039	F	0.021		0.0009	G	0.0028	<0.001	G	0.012	0.018	0.0040	F	0.0012	G	0.0051	E	0.0044	
	E	0.37		0.33	D	0.14	0.73	C	0.40	0.75	0.48	E	0.24	E	0.037	G	0.17	
013	E	n.m.		0.0016	F	n.m.	0.0007	F	n.m.	n.m.	<0.001	E	0.00028	G	0.00065	F	0.00069	
	D	0.68		0.73	D	4.6	0.096	F	0.20	0.39	0.22	E	0.19	F	0.60	D	0.44	
	D	<0.1		<0.1	E	<0.1	<0.1	F	<0.1	<0.1	<0.1	F	<0.10	E	<0.1	F	<0.1	
	H	3.9		<0.1	E	2.5	0.16	G	<0.1	<0.1	<0.1	H	<0.1	H	<0.1	G	<0.1	
	G	0.052		0.008	G	0.052	0.024	E	0.052	0.040	<0.01	F	0.009	G	0.058	D	<0.005	

01	G	0.0032	<0.0001	I	<0.005	0.0007	G	0.0027	<0.002	<0.005	D	<0.0005	G	<0.0005	G	<0.0005					
005	I	n.m.	<0.001	H	n.m.	<0.001	H	n.m.	0.035	<0.001	E	<0.001	H	<0.01	F	<0.01					
019	F	<0.001	0.0019	E	<0.0005	0.0022	E	0.0020	0.0026	0.0053	F	0.00014	I	0.0021	F	0.0014					
046	G	0.003	<0.0002	H	<0.005	0.0036	G	0.005	<0.005	0.0094	F	0.0005	I	0.036	D	<0.001					
	F	<0.1	<0.1	F	<0.1	<0.1	F	<0.1	<0.1	<0.1		<0.1	F	<0.1	F	<0.1					
026	E	0.0088	0.0030	F	0.0048	0.0017	F	0.0053	0.028	0.0016	F	0.00083	G	<0.00005	I	<0.0001					
026	G	0.002	<0.001	H	<0.002	<0.001	H	<0.001	0.012	0.0016	G	<0.0002	I	0.0032	F	<0.001					
0004	E	n.m.	<0.00001	F	n.m.	0.00006	F	n.m.	n.m.	0.00032	D	<0.00001	F	0.00032	E	0.00015					
0005	G	0.0071	<0.00005	G	n.m.	0.00011	E	n.m.	n.m.	0.00046	E	<0.00002	H	0.00021	E	0.00020					
0005	F	n.m.	<0.00005	F	n.m.	<0.00005	G	n.m.	n.m.	0.00027	E	0.00011	G	<0.00005	F	<0.00005					
0019	E	0.00022	0.00043	E	n.m.	<0.00005	G	n.m.	n.m.	0.00030	D	0.00009	F	<0.00005	F	<0.00005					
05	F	0.046	<0.005	F	0.027	<0.001	H	n.m.	<0.005	<0.001	H	<0.001	H	<0.005	F	<0.005					
0034	F	0.0076	0.00029	F	n.m.	0.0037	B	0.0027	0.0031	0.00002	H	0.00066	D	0.00092	D	<0.0005					
005	H	0.045	<0.001	G	n.m.	<0.001	G	n.m.	n.m.	0.0012	G	<0.005	H	0.014	E	0.0076					
00005	H	0.00033	<0.000005	H	n.m.	0.00001	G	n.m.	n.m.	0.00045	D	<0.000005	H	<0.000005	G	<0.00000					
0037	E	<0.0001	0.00009	E	<0.0001	<0.000005	I	<0.0001	<0.0001	0.00016	E	0.00017	E	0.00044	D	0.00038					
0028	G	0.012	0.0032	D	<0.005	0.0092	D	0.016	0.016	0.00012	G	0.0048	C	0.029	F	<0.005					
24		>55	37		>20	63		>32	>28	65		34		13		15					

ranfilter
chull
e to high background

Standard deviations: (Reproducibility)
A: ≦5% E: 50–100% I: >1000%
B: 5–10% F: 100–200%
C: 10–20% G: 200–500%
D: 20–50% H: 500–1000%

TABLE V

Concentration of elemental components and impurities in adhesives (in micrograms/gram of adhesive)

e	Uhu all adhesive	Uhu Paper fit	Uhu hard	Pelikam gum	Pelikanol white glue	Dekalin adhesive 1188	Ipacollan B 50 CS	Snap-Fix Photo Gum	Pattex	Pritt pin
	48	23	72	95	61.000	8.2	23	32	810	32.000
	420	<3.5	<8	20	—	<4	7.6	14	n.m.	n.m.
	<0.2	66	66	37	—	11	53	<0.15	n.m.	n.m.
	<0.2	<0.03	<0.07	3400	<900	0.65	0.70	18	<20	<200
	0.000018	0.00038	0.00078	<0.0003	0.0015	0.00058	0.00028	0.00047	<0.0005	0.0021
	<2	<2	<4	27	<2	<2	<2	<2	<2	<2
	0.020	0.052	0.15	0.34	0.018	0.039	0.18	0.0067	<0.013	0.041
	0.0067	0.071	0.17	1.6	—	0.047	0.061	0.075	n.m.	1.3
	0.28	2.1	3.4	1.9	1.5	3.3	4.7	0.50	3.6	1.5
	0.033	0.0030	0.0070	<0.0003	0.0081	0.0044	0.0017	0.017	0.037	0.25
	3.0	2.6	12	16	0.43	<0.4	1.8	230	2000	4.9
	n.m.	<0.02	<0.04	<0.03	<0.05	0.044	<0.02	0.0013	0.05	0.067
	2.5	0.75	2.0	1.9	6.0	1.3	7.5	0.13	230	<0.6
	<0.04	<0.04	<0.08	77	<0.04	<0.04	<0.04	<0.04	<0.04	<0.04
	<0.02	<0.04	<0.08	<0.05	<0.03	<0.04	<0.04	<0.03	<0.08	<0.03
	0.0023	0.18	0.42	0.22	<0.03	0.18	0.14	0.047	<0.05	0.82
	<0.002	<0.007	<0.015	0.0091	<0.005	<0.008	0.015	<0.006	<0.009	<0.03
	<0.005	<0.004	<0.008	<0.005	<0.03	<0.004	<0.004	<0.015	<0.008	<0.09
	<0.5	<0.7	<1.5	<1	<0.5	2.5	7.0	<0.5	<0.5	<0.5
	<0.0005	0.0005	2.3	1.1	<0.0005	<0.12	0.93	<0.0005	<0.0005	<0.0005

0.0023	<0.002	<0.004	0.030	0.00053	<0.002	0.0023	0.0047	0.004	0.15
0.070	0.022	0.10	0.051	<0.015	<0.004	0.022	0.087	<0.13	0.33
<0.002	<0.002	<0.004	0.032	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
<0.4	<0.4	<0.8	5.8	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4
0.00007	<0.0004	<0.0008	<0.0005	n.m.	<0.0004	<0.0004	0.00082	0.015	n.m.
0.010	0.0045	0.0054	0.0075	0.016	<0.0002	0.0063	0.0061	<0.002	<0.03
n.m.	0.0014	0.0018	0.0023	<0.0007	0.00045	0.0011	0.00073	<0.003	0.0011
<0.0002	0.0019	<0.00008	0.00095	<0.0002	<0.00004	<0.00005	0.00006	0.00082	0.0019
0.00013	<0.0001	<0.0001	<0.0001	0.0016	<0.0001	<0.0001	0.0028	<0.003	0.0056
0.00090	<0.001	<0.001	<0.001	0.0012	<0.001	<0.001	0.0034	<0.002	<0.005
<0.002	<0.002	<0.004	<0.003	<0.003	<0.002	<0.002	<0.003	<0.0008	<0.03
0.00090	<0.0001	<0.0001	<0.0001	<0.0006	<0.0001	<0.0001	<0.00015	0.0038	0.0059
0.073	0.018	0.036	<0.005	<0.12	<0.004	0.0055	0.029	<0.2	0.089
0.00025	<0.00004	0.00027	0.00037	0.00025	<0.00004	0.00043	0.00023	<0.00008	0.0045
0.0011	0.0011	0.0033	0.0014	0.00030	0.00028	0.0023	0.00026	0.00053	0.0040
0.0046	<0.002	0.024	<0.003	0.0016	<0.002	<0.002	0.0048	<0.005	0.00094
~470	~100	~170	~3700	~62.000	~32	~110	~290	~3100	~32.000

The total concentration of components which were activated was about 60 mg/filter. Therefore, glass fiber filters are unsuitable for use in activation analysis when activation products in the filter precipitates with half lives below about 40 hours are to be determined. The 70 cm^2-polyethylene foil contained the same components in similar amounts as most of the membrane filters used here. The predominant number of the elements identified were present in

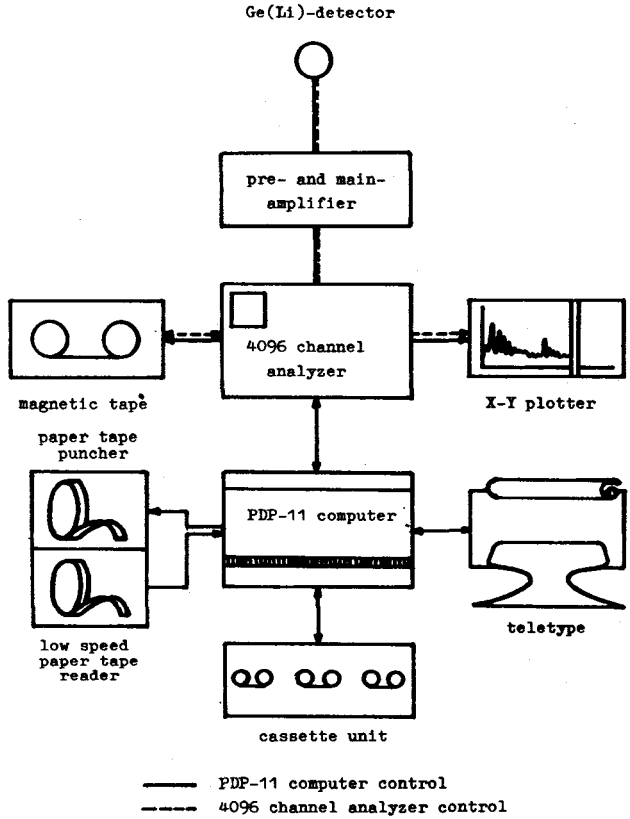

FIGURE 1 Schematic diagram of the computer controlled gamma spectrometer.

concentrations in the nanogram range and below. With the described measuring arrangement and 14 hours of counting time, radionuclides with half lives longer than about 100 days could be determined quantitatively, even in the picogram range.

It appeared that among the membrane filters those of polyamide and teflon yielded the lowest background activity and in addition, the structures

remained stable against neutron irradiation. Teflon filters with polyethylene gauze were partially stuck to their cover after irradiation. Among the membrane filters containing cellulose, those of cellulose acetate showed the lowest concentrations of activatable elements.

Adhesives

In the adhesives it was also possible to detect up to 30 elements (Table V). Dekalin, Elefantenkleber and Uhu-Papier-fit had the lowest concentration of activatable elements (30–100 μg/g of adhesive). After neutron irradiation most of the adhesives except Uhu-Papier-fit, Pelikanol glue, Dekalin adhesive and Snap Fix Photo Gum changed their colour to yellowish. Pattex even turned to a brown colour. The fastening of all adhesives was satisfactory with exception of the Pelikan gum that remained liquid. The most samples showed a variable number of bubbles, which might be attributed to gas formation of organic components.

For the purpose mentioned above Pelikanol glue and Pritt were eliminated due to their high Na content, Pelikan gum due to its high K content and Pattex and Snap Fix Photo Gum due to their high Zn content. Among the remaining adhesives Dekalin was chosen as most appropriate for the activation analysis of loaded cellulose filters because of its lowest content of activatable elements and because of the fact that it did not discolour during irradiation.

Detection limits and precision of filter precipitate analyses

When determining filter precipitates by activation analysis the detection limit for selected elements and the precision of the results depend mainly on the composition of the precipitate but also on the concentrations of the various elements in the blank filters, the adhesives and the polyethylene foil. For some pure element precipitates on cellulose acetate filters the following detection limits have been determined (in micrograms): Mn: 0.01, Zr:0.1, Cs:0.0005, Ba:0.01 and La:0.001.

Without background correction the precision of the results is better than 5% when the detected element masses lie at least a factor of 50 above the corresponding background concentrations. For the membrane filters examined in this work, this means that the element masses must be present in the filter precipitates at least in the microgram range.

If similar detection limits also for smaller concentrations are to be reached background corrections are necessary. However, one must consider the variation of composition of filter and adhesive materials for samples of different production batches. Therefore a careful determination of the background concentrations is always necessary.

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