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FULLER'S EARTH AS AN AGENT FOR PURIFYING HEAVY ORGANIC LIQUIDS*

WALLACE R. GRIFFITTS AND A. P. MARRANZINO, *U. S. Geological Survey, Denver, Colorado.*

For several years we have been investigating trace amounts of metals in various rocks and minerals. Bromoform, diiodomethane (methylene iodide), and acetylene tetrabromide were used to separate igneous rocks into their constituent minerals. Because, in some cases, we were determining low level metal content (1 or 2 ppm.) in mineral separates it became important to know if the liquids could pick up enough metal to contaminate the minerals, and, if so, how the metal might be removed. The concern deepened when our colleage A. P. Pierce told us that bromoform picked up important amounts of uranium, and J. C. Antweiler noted that

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bromoform became laden with lead when used to separate ore minerals. Antweiler and I. C. Frost found that as bromoform cools the crystals that form first contain very little lead and when separated and melted give a very clean liquid. In a search for a more convenient cleansing procedure we treated samples of organic heavy liquids with HCl-treated ion exchange resin (Amberlite IRA 400 and IRA 120 mixed in equal proportions) and also with fuller's earth. The fuller's earth had been used regularly to remove the tarry decomposition products that gradually darken bromoform and diiodomethane. Two fuller's earths from different sources gave equal results.

The treatment with fuller's earth is simple; shake a few grams of the earth with 100 to 150 ml. of liquid in a separatory funnel. After the earth rises to the top, draw off the clear liquid. Tapping of the funnel aids separation of the last liquid. Do not wash the clay with acetone or alcohol in an attempt to recover the heavy liquid. Tarry decomposition products and possibly metals trapped in the clay would thereby be washed out of the clay again and returned to the purified heavy liquid. The loss of heavy liquid can be minimized by keeping the amount of clay as small as possible. Ion exchange resins can be used in a similar manner and may involve less loss of liquid. The resins, however, are rather ineffective in bleaching the liquids.

Ten milliliters of each liquid were evaporated to dryness and the residues dissolved in nitric acid. Copper was determined in the solution with biquinoline and lead and zinc by dithizone. Analyses of the liquids before and after treatment gave the following results:

Sample number	Material	Treatment	Micrograms in 10 ml. sample		
			Pb	Cu	Zn
B1	Bromoform, from new bottle	None	1.2	.5	2
B2	Bromoform, from new bottle	Resin	1.	.5	<2
B3	Bromoform, from new bottle	Fuller's earth	1.	.5	<2
B4	Bromoform, used	None	< .4	1	<2
B5	Bromoform, used	Resin	< .4	< .5	<2
B6	Bromoform, used	Fuller's earth	< .4	< .5	<2
M1	Diiodomethane, used	None	*	*	4
M2	Diiodomethane, used	Resin	*	*	4
M3	Diiodomethane, used	Fuller's earth	*	*	<2
A1	Acetylene tetrabromide, used	None	2	2	<2
A2	Acetylene tetrabromide, used	Resin	.4	.5	<2
A3	Acetylene tetrabromide, used	Fuller's earth	< .2	< .5	<2

* Not determined because of strong interference from mercury, which F. N. Ward determined to be present in concentrations of about 450 ppm—or about 15,000 micrograms in 10 ml.

The treatment with fuller's earth effectively reduced the metal content of the liquids. The ion exchange resins were less effective. The low lead and zinc contents of the used bromoform probably result from the earlier bleaching by fuller's earth. The clay also removes dissolved water from bromoform, reducing the coagulation of suspended particles that sometimes takes place during separation of minerals finer than 200 mesh. The diiodomethane had been stabilized with mercury, which accounts for its high metal content. Stabilization with tin or copper may introduce similar amounts of these metals into the liquid. The metal that would cause the fewest problems in any investigation presumably should be used as a stabilizer.

The effectiveness of fuller's earth in removing metals from the liquids indicates that some clays may become seriously contaminated if they contact unclean heavy liquids. The contamination naturally is more serious if the amount of mineral in contact with the liquid is small, as in the later stages of purification of mineral concentrates, and may be critical if metal is selectively absorbed by some mineral.

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MEASUREMENT OF REFRACTIVE INDICES IN THIN SECTION

JOHN J. W. ROGERS, *Department of Geology, The Rice Institute, Houston, Texas.*

The uniform thickness and random orientation of grains in most thin sections are highly desirable features for many types of optical measurements. Unfortunately, however, the precise measurement of indices of permanently mounted material is difficult. One way to overcome this difficulty, while preserving the thin section, is to dissolve the Canada balsam mounting material around the edges by immersing the section in xylene. The solution process is easily watched and is stopped by washing the xylene out with acetone (which dries rapidly) when the xylene just reaches the outer edge of the rock slice (about 15 minutes for most sections). The section is then dried, and immersion oils are inserted under the edge of the cover glass and into contact with the rock. Indices of the various minerals are determined by using different oils sequentially and washing each of them out with acetone. Sections in which the rock slice is attached to the underlying slide by a mounting medium not easily soluble in xylene should have the outer edges of the rock "shaved" off before the cover glass is attached, and the cover should be cemented by Canada balsam. By removing edges which might be coated by an insoluble mounting medium, contact between immersion oil and mineral is assured.