



## FLOTATION ACTIVITY OF ALKYLAMINOETHANOLS AND THEIR XANTOGENATES

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### ABSTRACT

N-alkylated mono- and diethanolamines and their mono- and dixanthates have been used as reagent for flotation activity concentration of ores. Among the N-alkylated diethanolamines, and their mono- and dixantogenates were found effective collectors for enrichment of sulfide ores.

**Key words:** Flotation, Flotation reagents, Collectors, Ores, Diethanolamine, Alkyldiethanolamines, Xanthates.

### INTRODUCTION

Recently, the mining enterprises emerged in deterioration of processed ores. The processing of the minerals involved complex composition, characterized by a low content of valuable components, fine disseminated and similar physicochemical properties. To enrich the ores of this nature, the most common method of flotation is used, which have a decisive role in flotation activity reagents and process conditions<sup>1</sup>.

Flotation reagents used were costly as these are mainly from foreign production for processing polymetallic sulfide ores on Kazakhstan concentrators. This is one of the reasons for the low profitability of enterprises. There is an urgent need to create local-based reagents available in the Republic of Kazakhstan. Development of technology for the production of their own highly selective flotation reagents will reduce the cost of imports from other countries<sup>2</sup>.

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Creation of new and modification of conventional flotation reagents, as well as the establishment of the mechanism of their interaction with sulfide minerals will increase the degree of extraction of non-ferrous, precious and rare metals and greater energy-saving technologies for enrichment of ores.

An analysis of the scientific and technical literature on the flotation of sulfide ores shows that flotation activity is strongly dependent on the physicochemical properties and the surface activity of the various compounds of the chemical composition and structure<sup>3</sup>.

## **EXPERIMENTAL**

The synthesis of N-alkylated diethanolamines and their xanthates, conditions of interaction of various alkyl bromides with diethanolamine and analytical data of the synthesized compounds are reported by Erzhanov et al.<sup>4</sup> and Akimbaeva et al.<sup>5</sup>; xanthates (ANI-13, ANI-14, ANI-15, ANI-19) by Erzhanov et al.<sup>4</sup>, Akimbaeva et al.<sup>6</sup> and Anuarbekova<sup>7</sup>.

Flotation tests were carried out under laboratory conditions using a known reagent - butyl xanthate (Bx) and synthesized our's own new reagents (ANI-12, ANI-14) but without adding potassium cyanide as described in Fig. 1.

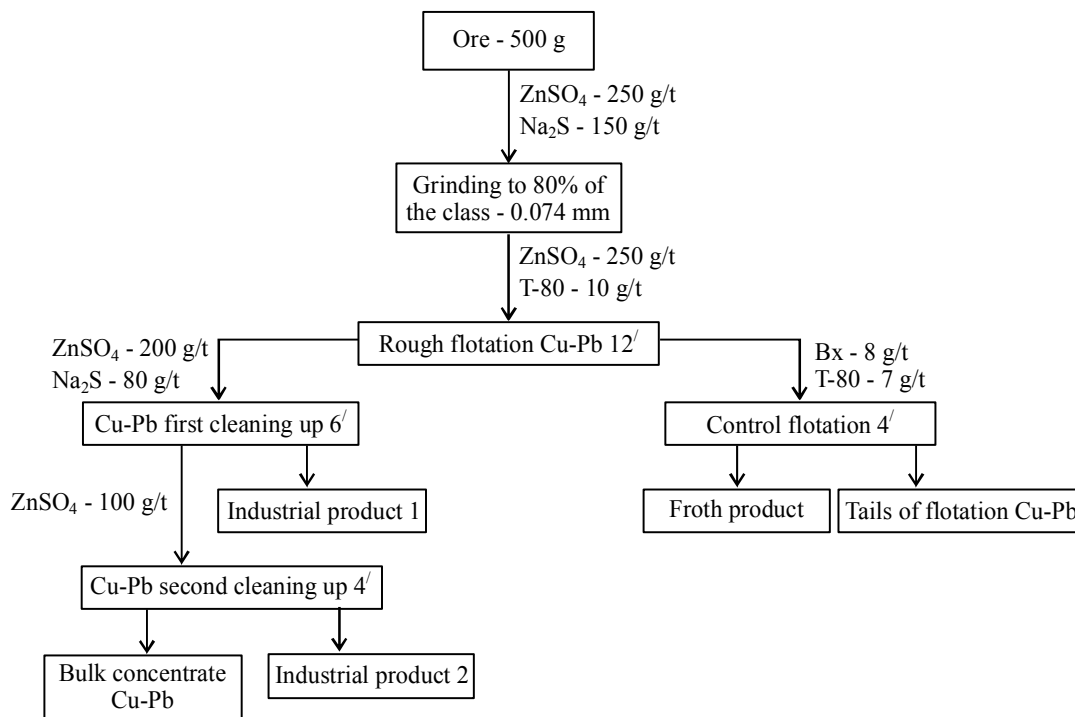
Basic copper-lead collective flotation was carried out for 10 min under conditions at which the collector and frother by fractional method (supplied).

The reagents used: Zn and Fe sulfide mineral depressants-ZnSO<sub>4</sub> and NaCN, collector-butyl xanthate (Bx), blowing agent-T-80, and new reactants contact synthesizing alkylated diethanolamine and xanthates.

## **RESULTS AND DISCUSSION**

Many flotation plants in Kazakhstan and the CIS as a collector used a mixture of collecting agents of different types or the same type but with different length of the nonpolar group: ethyl and butyl xanthates, xanthate and aeroflot, xanthate and a non-polar collector, a mixture of fatty acids, etc. The use of such mixtures is generally improves enrichment of complex ores<sup>8,9</sup>.

Experimental work on the extraction of a collective copper-lead concentrate and the establishment of optimal reagent regime needed to obtain low-order copper-lead or other metal concentrate was carried out by the scheme shown in Fig. 1.

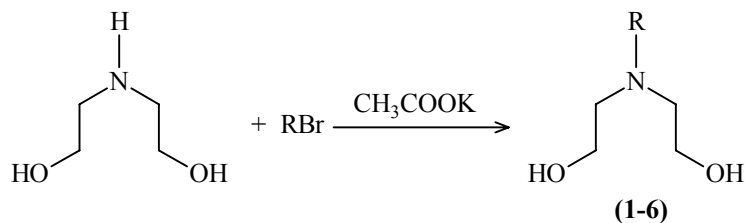


\* Note: - T-80, Ba - Blowing agents, Bx - Collector<sup>5</sup>

**Fig. 1: Schematic diagram and the reagent regime of copper lead collective flotation of Tishinsk field ore**

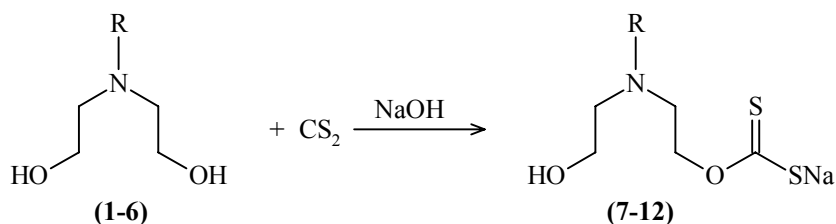
The laboratory of flotation reagents and enrichment of JSC "Center of the Earth Sciences, Metallurgy and Enrichment" studied flotation activity and carried out a comparative assessment of alkylethanolamines and xanthates synthesized by us (1-12). Experiments have shown that the substances N-(nonyl)-N-diethanolamine (ANI-12) (4) and N-(nonyl)-N-(2-hydroxyethyl)amino-2-ethylxanthate sodium (ANI-14) (9), which exhibit high flotation activity towards Zn, Pb, Ag, compared with the industrial flotation reagents and reagent comparison N-(tetrahydrocyclopentadienyl)-N',N''-dimethylurea (N,N-DU) (Table 1) among those represented interest.

Synthesis of alkyl diethanolamines (1-6) carried out by reacting diethanolamine and haloalkyls<sup>4,7</sup>, wherein the reagent source-diethanolamine is widely available commercial product, manufactured based on ethylene oxide and ammonia. The second starting material – nonyl bromide was also easily obtained by direct nonane halogenation or by addition of hydrogen halide to the 1-nonene.



R = -C<sub>6</sub>H<sub>13</sub> (**1**), R = -C<sub>7</sub>H<sub>15</sub> (**2**), R = -C<sub>8</sub>H<sub>17</sub> (**3**); R = -C<sub>9</sub>H<sub>19</sub> (**4**), R = -C<sub>10</sub>H<sub>21</sub> (**5**);  
R = -C<sub>12</sub>H<sub>25</sub> (**6**)

Reactions of the alkylated derivatives of diethanolamine **1-6** with carbon disulfide lead to sodium xanthates (**7-12**) in quantitative yields.



The structure of the synthesized compounds (**1-12**) set is based on an analysis of data of IR and <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy<sup>5-7,10-12</sup>.

As can be seen from Table 1, in dosage ANI-12-40 and 50 g/t Pb, recovery is 94.1, 90.1%, zinc and silver 90.6-91.5%. It should be noted that the flotation process was conducted without the addition of potassium cyanide and unsubstituted ethanolamine ANI-12 flotation activity is higher than its xanthate<sup>10</sup>.

Among dithiocarbonic derivatives of substituted ethanolamines, the compounds ANI-13 (N-octyl-N-bisethylxanthate sodium) (**13**)<sup>11</sup>, and ANI-19 (N-heptyl-N-2-hydroxyethylamino- 2-ethylxanthate sodium) (**8**)<sup>12</sup> showed high activity in the flotation enrichment of polymetallic ores of Tishinsk field. In determining of the flotation activity of the compounds ANI-13 and ANI-19, concentration was varied from 38 to 76 g/m.

We found that at a rate of ANI-19 76 g/t, compared with butyl xanthate solution, achieved high technological parameters for the flotation of copper and lead ore. The copper content using a new reagent-collector ANI-19 was 12.6% (basic mode-4.9%). Removing copper into bulk concentrate using ANI -19 was 83% (basic mode-79.8%). Removing zinc and pyrite in the tailings is increased to 84%.

**Table 1: Data flotation experiments using novel reagents (ANI-12, ANI-14) as a collectors**

| Reagents     | Dosage (g/t) | Products    | Output, (%) | Contents (%) |       |         | Recovery (%) |       |       |
|--------------|--------------|-------------|-------------|--------------|-------|---------|--------------|-------|-------|
|              |              |             |             | Pb           | Zn    | Ag. г/т | Pb           | Zn    | Ag    |
| Factory mode |              | concentrate | 6.17        | 22.08        | 20.48 | 260.7   | 87.9         | 82.6  | 82.5  |
|              |              | tailings    | 93.83       | 0.20         | 0.28  | 3.64    | 12.1         | 17.4  | 17.5  |
|              |              | nutrition   | 100.0       | 1.55         | 1.53  | 19.5    | 100.0        | 100.0 | 100.0 |
| N.N-DM       | 20           | concentrate | 5.97        | 23.60        | 21.51 | 286.0   | 90.9         | 85.6  | 87.55 |
|              |              | tailings    | 94.05       | 0.15         | 0.23  | 2.58    | 9.1          | 14.4  | 12.45 |
|              |              | nutrition   | 100.0       | 1.55         | 1.53  | 19.5    | 100.0        | 100.0 | 100.0 |
| ANI-12       | 40           | concentrate | 6.16        | 22.91        | 22.82 | 288.3   | 94.1         | 90.1  | 90.6  |
|              |              | tailings    | 93.84       | 0.09         | 0.16  | 1.73    | 5.9          | 9.9   | 9.4   |
|              |              | nutrition   | 100.0       | 1.50         | 1.56  | 19.6    | 100.0        | 100.0 | 100.0 |
| ANI-12       | 50           | concentrate | 6.24        | 22.80        | 22.31 | 283.9   | 94.2         | 90.4  | 91.5  |
|              |              | tailings    | 93.76       | 0.09         | 0.16  | 1.69    | 5.8          | 9.6   | 8.5   |
|              |              | nutrition   | 100.0       | 1.51         | 1.54  | 19.3    | 100.0        | 100.0 | 100.0 |
| ANI-14       | 40           | concentrate | 4.15        | 15.31        | 18.80 | 289.2   | 83.7         | 81.9  | 71.2  |
|              |              | tailings    | 95.85       | 4.10         | 2.17  | 1.61    | 16.3         | 18.1  | 28.8  |
|              |              | nutrition   | 100.0       | 1.53         | 1.56  | 19.5    | 100.0        | 100.0 | 100.0 |
| ANI-14       | 50           | concentrate | 4.20        | 16.42        | 17.53 | 287.2   | 84.3         | 81.3  | 81.8  |
|              |              | tailings    | 95.80       | 2.09         | 2.14  | 1.49    | 15.7         | 18.7  | 18.2  |
|              |              | nutrition   | 100.0       | 1.54         | 1.53  | 19.4    | 100.0        | 100.0 | 100.0 |

In addition to these studies, it was found that the substances ANI-13 and ANI-19 as an additional flotation reagent into butyl xanthate (Bx) show high activity in the flotation enrichment of polymetallic ores. These data are confirmed by experiments carried out at the factory and are shown in Table 2. In the laboratory, it was shown that ANI-13 and ANI-19 can also be used as additional flotation reagent for the enrichment of gold rocks.

**Table 2: Flotation activity of the xanthates ANI-13 and ANI-19**

| Title products                  | Output (%) | Contents (%) |             |            |      | Recovery (%) |             |             |             | Note   |
|---------------------------------|------------|--------------|-------------|------------|------|--------------|-------------|-------------|-------------|--------|
|                                 |            | Cu           | Pb          | Zn         | Fe   | Cu           | Pb          | Zn          | Fe          |        |
| Basic reagent regime for 1000 g |            |              |             |            |      |              |             |             |             |        |
| Cu-Pb concentrate               | 9.3        | <b>4.9</b>   | <b>5.8</b>  | <b>7.2</b> | 33.4 | <b>79.8</b>  | <b>75.5</b> | 13.5        | 53.7        |        |
| Industrial product 2            | 1.8        | 0.6          | 1.25        | 10.4       | 23.2 | 1.9          | 3.2         | 3.8         | 7.2         | Bx     |
| Industrial products 1           | 6.9        | 0.3          | 0.9         | 8.7        | 11.0 | 3.6          | 8.7         | 12.1        | 13.1        | 76 g/T |
| foam control                    | 2.0        | 0.2          | 0.5         | 8.6        | 6.97 | 0.7          | 1.4         | 3.5         | 2.4         | 54 g/T |
| tailings                        | 80.0       | 0.1          | 0.1         | 4.15       | 1.7  | 14.0         | 11.2        | <b>67.1</b> | <b>23.5</b> |        |
| original ore                    | 100        | 0.57         | 0.71        | 4.95       | 5.87 | 100          | 100         | 100         | 100         |        |
| Cu-Pb concentrate               | 7.8        | <b>5.05</b>  | <b>1.2</b>  | <b>8.2</b> | 33.4 | <b>77.0</b>  | <b>40.7</b> | 12.7        | 44.8        |        |
| Industrial product 2            | 1.17       | 0.6          | 1.0         | 8.2        | 24.7 | 1.4          | 5.1         | 1.9         | 5.0         | Bx     |
| Industrial products 1           | 7.9        | 0.3          | 0.4         | 7.2        | 13.9 | 4.6          | 13.8        | 11.3        | 18.9        | ANI-13 |
| foam control                    | 1.95       | 0.3          | 0.6         | 8.1        | 10.4 | 1.1          | 5.1         | 3.1         | 3.5         | 38 g/T |
| tailings                        | 81.18      | 0.1          | 0.1         | 4.4        | 2.0  | 15.9         | 35.3        | <b>71.0</b> | <b>27.9</b> | ANI-13 |
| original ore                    | 100        | 0.51         | 0.23        | 5.03       | 5.82 | 100          | 100         | 100         | 100         | 38 g/T |
| Cu-Pb concentrate               | 3.66       | <b>8.7</b>   | <b>11.8</b> | <b>9.6</b> | 13.1 | <b>64.6</b>  | <b>71.8</b> | 7.1         | 8.2         | 54 g/T |
| Industrial product 2            | 1.5        | 2.0          | 2.5         | 7.95       | 5.8  | 6.1          | 6.2         | 2.4         | 1.5         | ANI-13 |
| Industrial products 1           | 3.82       | 0.86         | 0.8         | 5.2        | 5.8  | 6.7          | 5.1         | 4.0         | 3.8         | 76 g/T |
| foam control                    | 2.1        | 1.1          | 0.6         | 5.5        | 6.4  | 4.7          | 2.1         | 2.3         | 2.3         | T-80   |
| Tailings                        | 88.92      | 0.1          | 0.1         | 4.7        | 5.5  | 18.0         | 14.8        | <b>84.2</b> | <b>84.1</b> | 54 g/T |
| original ore                    | 100        | 0.49         | 0.60        | 4.96       | 5.81 | 100          | 100         | 100         | 100         |        |

Cont...

| Title products        | Output (%) | Contents (%) |            |             |      | Recovery (%) |             |             |             | Note           |
|-----------------------|------------|--------------|------------|-------------|------|--------------|-------------|-------------|-------------|----------------|
|                       |            | Cu           | Pb         | Zn          | Fe   | Cu           | Pb          | Zn          | Fe          |                |
| Cu-Pb concentrate     | 2.35       | <b>12.6</b>  | <b>4.9</b> | <b>10.1</b> | 16.3 | <b>83.0</b>  | <b>49.2</b> | 4.8         | 6.6         |                |
| Industrial product 2  | 0.7        | 1.74         | 3.78       | 8.9         | 8.7  | 3.4          | 11.3        | 1.3         | 1.1         | ANI-19         |
| Industrial products 1 | 2.8        | 0.72         | 1.3        | 6.75        | 7.3  | 5.7          | 15.6        | 3.8         | 3.5         | 76 g/T<br>T-80 |
| foam control          | 6.81       | 0.16         | 0.18       | 6.3         | 10.2 | 3.1          | 5.2         | 8.7         | 12.0        | 54 g/T         |
| tailings              | 87.34      | 0.02         | 0.05       | 4.6         | 5.1  | 4.9          | 18.7        | <b>81.4</b> | <b>76.8</b> |                |
| original ore          | 100        | 0.36         | 0.23       | 4.94        | 5.8  | 100          | 100         | 100         | 100         |                |

Thus, the results of experiments on the high flotation cycle demonstrated the possibility of using new collector-ANI-19 (N-heptyl-N-2-hydroxyethylamino-2-ethylxanthate sodium) (**8**) for enrichment of sulfide ores. An increase in the extraction of copper in the copper-lead concentrate by 3.2% and increase in the content of copper in the copper-lead concentrate by 7.7% were marked. ANI-13 (N-octyl-N-bisethylxanthate sodium) (**13**) and ANI-19 (**8**) may also be used as additional flotation reagent in the beneficiation of gold species.

## CONCLUSION

- (i) N-C6-C10, and C12-alkyl diethanolamines and their mono- and dithioxanthates were synthesized.
- (ii) It is established that the mono- and dithioxanthates of alkylsubstituted diethanolamines ANI-13 and ANI-19 exhibit high flotation activity in the enrichment of polymetallic ores.
- (iii) High flotation activity of xanthates ANI-13 and ANI-19 was confirmed by the results of factory tests carried on Tishinsky mine ores.
- (iv) Tests of ANI-13 and ANI-19, held in Balazhalsk gold deposit shows that they are not inferior to industrial flotation reagent SWIM.
- (v) Flotation reagents as ANI-12 and ANI-14 have high flotation activity - more than 90%, based on lead, zinc and silver.

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