No evidence for either of these suggestions has been found in the present work. The structure refinement, density measurement and chemical composition all indicate that the 'molecule' is not $MoAl_{13}$ but $MoAl_{12}$.

In all three structures the transition-metal to aluminum distance is significantly shorter than the inter-aluminum distance, which suggests that the strongest interaction is between aluminum and transition-metal atoms. This shortening of these bonds is more marked in $MoAl_{12}$ than in the other Mo–Al phases, with the exception of $MoAl_4$ (Leake, private communication), but is not so marked as in the phases of aluminum with transition metals of the first long period (Taylor, 1954).

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Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.

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The empirical formulae of intermetallic compounds by neutron activation.

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Neutron activation analysis has been applied to the determination of the empirical formulae of some technetium–aluminum and molybdenum–aluminum compounds. An outline of the technique is given, as this method is of general application and is particularly suitable when accurate results are required from limited quantities (*ca.* 5 mg) of material.

In neutron activation analysis (see *e.g.* Jenkins & Smales, 1956), the sample for analysis is irradiated with neutrons together with a known standard of the element to be determined. The induced activities for each constituent element can be characterized by the natures and energies of emitted radiations and their half-lives. When the measured radioactivity is of short half-life, it is usual for the analysis to be non-destructive, using some form of γ-ray spectrometry.

Several advantages are offered:

(i) It is highly sensitive, which means that only small quantities of material are required. Satisfactory results can be obtained with one milligram or less.

(ii) It is specific for the element determined, provided that elementary precautions are taken to ensure measurement of radioactivity which is characteristic of that element. Gamma-ray spectrometry and half-life determinations are often sufficient for this.

(iii) Where the activity is short-lived, repeat analyses can be carried out on the same sample to improve precision.

When the specimen contains nuclides which absorb neutrons strongly, errors can arise if the distributions of these elements in the specimen and sample differ. This is because the full neutron flux is unable to reach the inner regions of the solid sample. This source of error can usually be overcome by dispersing the material in a medium with a low neutron cross-section (*e.g.* in aqueous solution). The method is then destructive to the sample, but repeated measurements can still be made on the solution, if the half-lives are suitable.

Examples of analyses

Some doubt about the true composition of the intermetallic compound '$MoAl_{12}$' has resulted from the conventional chemical analyses reported by Clare (1960). These analyses indicate a deficiency of molybdenum and an average composition corresponding to $MoAl_{13}$, although

Table 1. *The analysis of the intermetallic compounds* '$TcAl_{12}$' *and* '$MoAl_{12}$'

		Sample wt.	Tc found	Al found	Al/Tc	Mean Al/Tc	$\frac{(Al+Tc-Sample)}{Sample}\times 100$
'$TcAl_{12}$'	(1)	1·0060 mg	0·246 mg	0·779 mg	11·66	11·80	+1·8
			0·232	0·756	11·94		−1·8
	(2)	0·5834	0·137	0·452	12·09	11·80	+0·9
			0·140	0·439	11·50		−0·7
			Mo found	Al found	Al/Mo	Mean Al/Mo	$\frac{(Al+Mo-Sample)}{Sample}\times 100$
'$MoAl_{12}$'	(3)	2·566 mg	0·580 mg	1·87 mg	11·80	11·7	−4·7
			0·575	1·92	11·87		−2·7
	(4)	2·143	0·482	1·67	12·30	12·2	+0·5
			0·487	1·66	12·12		+0·5

only the molybdenum content was measured. However, an X-ray structure refinement by Walford (1964) (p. 57 of this issue) failed to detect any deviation from the composition Mo:Al = 1:12. Very limited quantities of extracted single crystals of '$MoAl_{12}$' and the isomorphous '$TcAl_{12}$' were available; results obtained by neutron activation analysis have allowed a choice to be made between these conflicting results.

The Tc:Al ratio was determined by use of the reactions

$^{99}Tc\ (n\gamma)\ ^{100}Tc$ (half-life 16 sec).
Energy (γ) = 0·54 meV

$^{27}Al\ (n\gamma)\ ^{28}Al$ (half-life 2·3 min).
Energy (γ) = 1·78 meV.

Specimens were weighed on a deci-microbalance and sealed in clean polythene containers for irradiation. Weighing provided a check on the total weight of the two constituents, which were determined individually. Aluminum standards were prepared in a similar manner, using freshly cut pieces of 99·99% pure aluminum. Technetium standards were made by weighing out appropriate amounts of an ammonium pertechnetate solution.

The respective γ-rays were measured with a scintillation counter after irradiation in the Harwell reactor BEPO. A pneumatic device which transferred the samples rapidly from reactor to laboratory allowed counting to be started within 20 seconds of irradiation. For technetium, which was determined first because of its short half-life, a single-channel spectrometer was set to select the 0·54 meV γ-ray after irradiation for 3 seconds. Aluminum was determined by counting all γ-rays of energy greater than 1·75 meV after irradiation for 20 seconds. In both cases, decay curves were plotted from the results of alternate counts on sample and standard.

The Mo:Al ratio was determined using the reactions

$^{27}Al\ (n\gamma)\ ^{28}Al$ (half-life 2·3 min).
Energy (γ) 1·78 meV

$^{98}Mo\ (n\gamma)\ ^{99}Mo \xrightarrow{\beta} {}^{99m}Tc$ (half-life 6 hr).
Energy (γ) 0·14 meV.

The aluminum was determined in the same way as in Tc:Al.

Molybdenum was determined by irradiating samples with standards of ammonium molybdate for 30 minutes, allowing a two-day delay, and then counting the 0·14 meV γ-ray due to ^{99m}Tc with a 100-channel kicksorter.

The results of these analyses are summarized in Table 1.

A check on the overall accuracy of determination is provided by summing the weights of the constituents as determined by neutron activation analysis and comparing these with the directly measured sample weight. Differences are listed in the last column of the table as percentages of the total sample weight.

Sample (4) was dissolved and irradiated as an aqueous nitrate solution. The results by this method are more reliable.

A full account of the techniques will be published.

Conclusions

Although there is some spread in the results they are sufficiently consistent to indicate probable 'crystallographic' formulae of $MoAl_{12}$ and $TcAl_{12}$ with milligram quantities of material. Refinements of technique should make it possible to obtain equally satisfactory results with single crystals in the 10 μg range.

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