FILE NAME: Johnson & Johnson (JAJ)

DATE: 0000

DOC#: JAJ217

DOCUMENT DESCRIPTION: Report - Concentration of Amphibole and Serpentine asbestos from Talc by froth flotation

CONCENTRATION OF AMPHIBOLE AND SERPENTINE ASBESTOS FROM TALC BY FROTH FLOTATION

Protected Document--Subject to Protective Order

TABLE 1: Flotation Results of 0.5% Asbestos Sample

	Test No.	Sample No.	Fraction	Fe p.p.m.	% Crocidolite	% Recovery	K
A	1	1	Conc.	0.0	0.0		
		2	Tailing	8.4	6.0	100	12
	2	3	Conc. 1	0.0	0.0		
		4	Conc. 2	0.0	0.0	(0.5% Crocidolite)	
		5	Conc. 3	0.0	0.0		
		6	Conc. 4	0.0	0.0		
		7	Middling	2.16	1.5		
		8	Tailing	12.6	8.5	91.0	17
B	3	9	Conc.	0.0	0.0		
		10	Tailing	8.0	8.0	100	12
	4	11	Conc. 1	0.0	0.0		
		12	Conc. 2	0.0	0.0		
		13	Conc. 3	0.0	0.0	(0.5% Crocidolite + 95% Chrysotile)	
		14	Conc. 4	0.0	0.0		
		15	Middling	2.0	1.5		
		16	Tailing	13.1	9.0	90.0	18
				Mg p.p.m.	% Chrysotile		
C	5	17	Conc.	0.0	0.0		
		18	Tailing	1.18	8.5	100	17
	6	19	Conc. 1	0.0	0.0		
		20	Conc. 2	0.0	0.0	(0.5% Chrysotile)	
		21	Conc. 3	0.0	0.0		
		22	Conc. 4	0.0	0.0		
		23	Middling	0.30	2.0		
		24	Tailing	1.70	13.0	90	26

Protected Document--Subject to Protective Order

examination and the X-ray tube was operated at 30 KV and 20 ma. A diffractometer scan rate of 1° 2θ per minute was employed and the inlet and outlet slits were $\frac{1}{2}^{\circ}$ 2θ and 0.2 mm respectively. Diffractometer traces of Italian talc, chrysotile, crocidolite and actinolite used in the separation experiments are given by Figs. 6 - 9.

Results

The results of a number of tests upon a talc sample containing 0.5% chrysotile and 0.5% crocidolite by weight and also a sample containing both crocidolite and chrysotile in 0.5% portions are given in Table 1. In Table 1 test No. 1 represents a single stage flotation separation of crocidolite and talc. It can be seen that the concentration of crocidolite in the tailings is 6% by weight, no crocidolite being detected in the floated talc. Test No. 2 in Table 1 represents a two stage separation of crocidolite and talc. In the two stage separation the tailings fraction from four single stage tests have been combined together and retreated to give a final tailings and a secondary flotation product referred to as middlings. By employing two stages of separation it can be seen that the final tailings is richer in asbestos than a single stage flotation tailings. However, some asbestos is lost in the middlings fraction. Tests 3 and 4 in Table 1 show the results of the crocidolite separation from the mixture of crocidolite and chrysotile. Again a 6% tailings assay in a single step and a 9.0% assay for crocidolite in a two stage separation. The chrysotile did not therefore interfere with the crocidolite separa-

Protected Document--Subject to Protective Order

TABLE 2: Flotation Results of 1% Asbestos Sample

	Test No.	Sample No.	Fraction	Fe p.p.m.	% Crocidolite	% Recovery	Concentration Ratio K
A	7	25	Conc.	0.30	0.25		
		26	Tailing	30.60	21.00	75.90	21
	8	27	Conc.	0.40	0.25		1% Crocidolite
		28	Middling	9.60	7.00		
		29	Tailing	55.60	39.00	81.25	39
B	9	30	Conc.	0.10	0.00	0.00	
		31	Tailing	23.60	16.00	100.00	
	10	32	Conc.	0.00	0.00		1% Crocidolite + 1% Chrysotile
		33	Middling	11.60	8.00		
		34	Tailing	46.60	33.00	66.00	33
				Mg p.p.m.	% Chrysotile		
B	9	30	Conc.	0.00	0.00		
		31	Tailing	2.70	21.00	100.00	21
	10	32	Conc.	0.00	0.00		1% Crocidolite + 1% Chrysotile
		33	Middling	0.50	4.00		
		34	Tailing	4.80	37.50	90.62	37.50
C	11	35	Conc.	0.00	0.00		
		36	Tailing	2.60	20.00	100.00	20
	12	37	Conc.	0.00	0.00		1% Chrysotile
		38	Middling	0.60	4.50		
		39	Tailing	5.10	40.00	87.32	40

Protected Document--Subject to Protective Order

TABLE 3: Flotation Results of 2% Asbestos Samples

	Test No.	Sample No.	Fraction	Fe p.p.m.	% Crocidolite	% Recovery	Concentration Ratio K
A	13	40	Conc.	0.30	0.25		
		41	Tailing	37.60	26.00	88.35	13
	14	42	Conc.	0.00	0.00		2% Crocidolite
		43	Middling	8.60	6.00		
		44	Tailing	74.60	52.00	86.96	26
B	15	45	Conc.	0.30	0.25		
		46	Tailing	39.60	28.00	89.09	14
	16	47	Conc.	0.00	0.00		2% Crocidolite + 2% Chrysotil
		48	Middling	6.60	5.00		
		49	Tailing	61.60	43.00	92.95	21.5
				Mg p.p.m.	% Chrysotile		
B	15	45	Conc.	0.00	0.00		
		46	Tailing	3.50	27.00	100.00	13.5
	16	47	Conc.	0.00	0.00		2% Crocidolite + 2% Chrysotil
		48	Middling	0.80	6.00		
		49	Tailing	5.15	40.30	91.38	20.15
C	17	50	Conc.	0.00	0.00		
		51	Tailing	3.50	27.00	100.00	13.5
	18	52	Conc.	0.00	0.00		2% Chrysotile
		53	Middling	1.70	13.00		
		54	Tailing	5.25	41.00	75.93	20.5

Protected Document--Subject to Protective Order

tion. Tests 5 and 6 in Table 1 show the results obtained from the separation of a 0.5% chrysotile/talc mixture. In Test 5 the tailings were found to contain 8.5% chrysotile while test 6 shows that the tailings produced from a two stage separation assayed 13.0% chrysotile by weight a considerable upgrading from the original 0.5%.

Table 2 contains similar results to Table 1 except all the tests were performed with 1% additions of crocidolite and chrysotile and a 1% crocidolite and chrysotile mixture. From test 7 in Table 2 we see that in a single step a tailings containing 21% crocidolite was produced while in a single step for chrysotile (test 11) tailings with 20% chrysotile was obtained. The two stage separation results for crocidolite and chrysotile given by tests 8 and 12 respectively show an improvement to 39% and 40% as compared with 29% and 20% in the single stage tests.

Table 3 contains the results obtained from the separation of a series of samples containing 2% crocidolite and chrysotile respectively and also the results from a 2% crocidolite and chrysotile mixture. In a single stage separation for crocidolite (test 3) a tailings containing 26% crocidolite was obtained while a tailings containing 52% crocidolite was obtained in a two stage separation (test 4). For chrysotile, tailings fractions containing 27% and 40.3% chrysotile for single and two stage separations respectively were obtained (tests 15 and 16). The results for the 2% crocidolite, 2% chrysotile in talc samples gave 28% and 43% for crocidolite in the tailings fractions from single and two stage separations and 27%

Protected Document--Subject to Protective Order

TABLE 4 (Continued)

Test No.	Sample No.	Fraction	% Crocidolite		% Chrysotile		Remarks
			A.A.S.	X.R.D.	A.A.S.	X.R.D.	
13	40	Conc.	0.25	0.00			2% Crocidolite
	41	Tailings	26.00	30.00			
14	42	Conc.	0.00	0.00			
	43	Middlings	6.00	7.00			
	44	Tailings	52.00	54.00			
15	45	Conc.	0.25	0.00			2% Crocidolite 2% Chrysotile
	46	Tailings	28.00	29.00			
16	47	Conc.	0.00	0.00			
	48	Middlings	5.00	7.00			
	49	Tailings	43.00	43.00			
15	45	Conc.			0.00	0.00	2% Crocidolite 2% Chrysotile
	46	Tailings			27.00	29.00	
16	47	Conc.			0.00	0.00	
	48	Middlings			6.00	7.00	
	49	Tailings			40.30	38.00	
17	50	Conc.			0.00	0.00	2% Chrysotile
	51	Tailings			27.00	30.00	
18	52	Conc.			0.00	0.00	
	53	Middlings			13.00	12.00	
	54	Tailings			41.00	43.00	

Protected Document--Subject to Protective Order

TABLE 4: Comparison of the analysis of flotation products by X-ray Diffraction and wet chemical analysis

Test No.	Sample No.	Fraction	% Crocidolite		% Chrysotile		Remarks
			A.A.S.	X.R.D.	A.A.S.	X.R.D.	
7	25	Conc.	0.25	0.00			
	26	Tailings	21.00	23.00			
8	27	Conc.	0.25	0.00			
	28	Middlings	7.00	6.00			1% Crocidolite
	29	Tailings	39.00	40.00			
9	30	Conc.	0.00	0.00			
	31	Tailings	16.00	19.00			1% Crocidolite 1% Chrysotile
10	32	Conc.	0.00	0.00			
	33	Middlings	8.00	7.00			
	34	Tailings	33.00	36.00			
9	30	Conc.			0.00	0.00	
	31	Tailings			21.00	20.00	1% Crocidolite 1% Chrysotile
10	32	Conc.			0.00	0.00	
	33	Middling			4.00	5.00	
	34	Tailings			37.50	35.00	
11	35	Conc.			0.00	0.00	
	36	Tailings			20.00	19.00	1% Chrysotile
12	37	Conc.			0.00	0.00	
	38	Middlings			4.50	6.00	
	39	Tailings			40.00	41.00	

Protected Document--Subject to Protective Order

CONCENTRATION OF AMPHIBOLE AND SERPENTINE ASBESTOS FROM TALC BY FROTH FLOTATION.

INTRODUCTION.

A great deal of concern surrounds the possible asbestos content of talc powders used in cosmetic, pharmacutical and other industrial and consumer products. Asbestos minerals are known to be associated with the increased incidence of certain types of cancer and it would be of some industrial importance if it were possible to reduce the level of asbestos minerals in a talc powder to an insignificant level with respect to their possible health hazard. Froth flotation is a technique used widely to separate fine mineral particles and over 90% of the world's non ferrous minerals are produced from crushed and finely ground rock using this technique. This concentration technique has been applied to the concentration of commercial and non commercial forms of asbestos minerals from talc. It has been shown to be very efficient in reducing the asbestos content of a powder and producing a residue (which is referred to in mineral separation terms as a 'tailings' in which the asbestos minerals can be easily detected and quantified.

The technique has therefore a second significant use in the detection and quantification of asbestos minerals in talc powder samples. Most quantiative mineralogical techniques are only capable of detecting asbestos mineral impurities in talc powders down to the

Protected Document--Subject to Protective Order

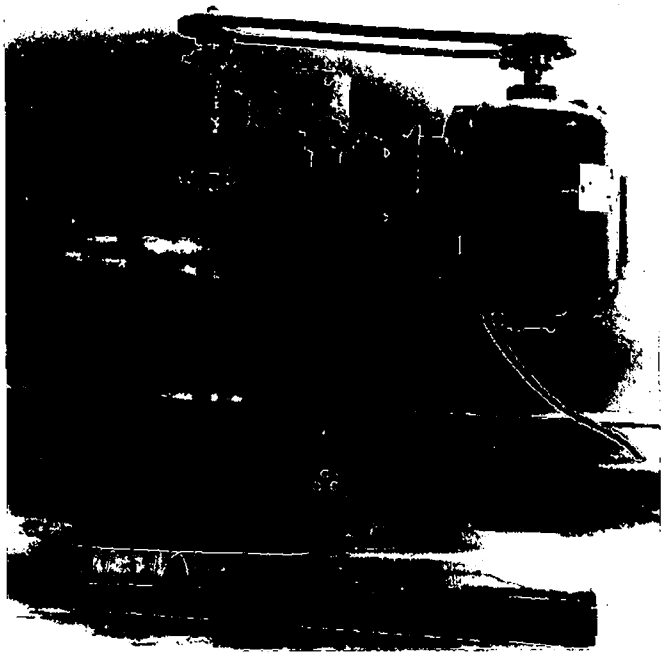


FIG. 1: Laboratory Flotation Cell

Protected Document--Subject to Protective Order

1% by weight level. These techniques include X-ray diffractometry, differential thermal analysis and ~~improved~~ infrared analysis. Of these techniques X-ray diffraction would appear to be the most useful. It allows both a positive identification of the mineral phase to be made and also an accurate determination of mass fraction by comparison of the relative intensity of the asbestos mineral reflections from the powders being tested to those of a set of standard reflections obtained from talc powder samples spiked with various quantities of different asbestos minerals.

The use of froth flotation to concentrate asbestos minerals from talc is so efficient that their detection by the X-ray diffraction method is accurate to an order or magnitude lower than in the untreated powder, i.e.: 0.1% or less. Efficient separation or 'pre-concentration depends upon the selection of the most amenable chemical conditions under which the flotation separation should be performed. The separation is made in an aqueous environment in which both talc and asbestos are in suspension. One of the minerals has to be hydrophobic or made hydrophobic in order to remove it from the aqueous suspension using air bubbles. Talc has a well known natural hydrophobicity and can be improved however by use of suitable cationic chemicals e.g.: amines, which selectively adsorb onto the talc particles and improve their hydrophobic character. Asbestos minerals have no natural hydrophobic character, therefore no natural floatability and will remain in the froth flotation cell while talc is being floated. Some care however has to

Protected Document--Subject to Protective Order

be exercised in the choice of frothing agent (which is used to produce a froth at the top of the flotation cell in which the floated mineral is held and removed) to ensure that it does not impart hydrophobic properties to any asbestos mineral particles. It must also produce a froth which drains easily removing as low an amount of water as possible from the cell in which asbestos mineral particles may be in suspension.

The pH of the aqueous medium is also of importance in the separation, as this can also affect the efficiency of the flotation by interfering with the properties of the frothing agant and also influencing the natural floatability of the talc and asbestos minerals. The pH control also produces a non flocculated system in which particles of asbestos and talc are separated in suspension. The control of pH is also desirable when a chemical is used to increase the hydrophobic nature of the talc as it can easily be adsorbed onto the asbestos minerals as well. Selection of the pH to produce minimum adsorption of the chemical onto the asbestos should therefore be made.

METHODS MATERIALS AND APPARATUS.

The froth flotation tests reported here were all performed in a modified 'Knapp and Bates' flotation cell with a capacity of 3 litres of mineral suspension. An impeller in the flotation cell keeps the mineral in suspension and also draws in air for flotation purposes. It then mixes the air with the mineral suspension to

Protected Document--Subject to Protective Order

JNJAZ55_000007958

promote rapid adhesion of particles to air bubbles; the impeller was operated at 1500 r.p.m. The amount of air drawn into the cell was approximately 17 litres/minute. The flotation cell used is illustrated by Fig: 1.

The minerals used for experimentation were as follows:

1) Italian talc, 00000 grade supplied by Zach Cartwright Ltd. Its general formulae $Mg_6\ Si_8\ O_{20}\ (OH)_4$.

2) Serpentine asbestos, (chrysotile) which is the most common of the asbestiform minerals. It accounts for more than 95% of the asbestos fibre produced today. Its general formulae is $Mg_{12}\ Si_8\ O_{20}\ (OH)_2$. Like talc it is a sheet silicate which occurs as very fine rolled sheets of magnesium silicate.

3) Amphibole asbestos, two varieties of which were used these were

Crocidolite $Na_2Fe_3^{++}Fe_2^{+++}Si_8O_{22}(OH)_2$

Actinolite $Ca_2(MgFe^{++})_5Si_8O_{22}(OH)_2$

The chrysotile and crocidolite samples were supplied by the M.R.C. Research Unit, Penarth, Glam, as being representative of all commercial asbestiform minerals of the serpentine and amphibole varieties.

Protected Document--Subject to Protective Order

The Actinolite sample was included to represent the type of amphibole more likely to be found associated with talc. The actinolite itself came from the same mine as the talc used in the experimentation but was not found in association with the talc. It occurred with other minerals and before addition to the talc in a powdered form it was concentrated into a pure fraction using a Franz isodynamic magnetic separator.

The chrysotile and crocidolite asbestos were already in a very finely powdered form and required no size reduction before addition to the Italian talc for separation experiments. The actinolite was however first ground to a size similar to that of the talc before addition.

Ordinary tap water was used in the flotation tests and the pH of the suspensions was adjusted using sulphuric acid and sodium hydroxide. The frothing agent used was a commercial product Aerofroth 65 manufactured by Cyanmide Ltd., it is essentially a poly-propylene-glycol of high molecular weight which is totally soluble in water and is therefore easy to add to the flotation cell. It was found that a suitable concentration of 1.5 ml of a 5% weight/volume solution of the frothing agent gave an easily managed froth removing very little liquid from the flotation cell.

Flotation separation tests were performed with talc samples in which asbestos was added to represent 0.5%, 1.0% and 2.0% asbestos by weight. A series of tests with

Protected Document--Subject to Protective Order

a mixture of amphibole and serpentine asbestos was also performed to investigate the concentrating action in the presence of two forms of asbestos. The talc samples were weighed and added to the flotation cell and mixed thoroughly with water the weighed asbestos sample was then added and also mixed thoroughly into the talc suspension. Care was taken to ensure the talc was completely wetted ~~melted~~ by mixing thoroughly, no wetting agents were used. When the talc/asbestos mixture was considered to be well mixed the frothing agent was added and pH adjustment made. The air inlet to the cell was then opened and the floated talc removed from the top of the cell with a scraper. The test was run until no visible talc was found to be reporting to the top of the cell, care was taken to ensure no talc froth was sticking to the cell sides which might contaminate the asbestos material remaining in the cell as tailings.

From extensive tests the most advantageous conditions for advantageous conditions for asbestos talc separation was found to be as follows:

Water in the cell 3 litres
pH adjusted to 2.0 with sulphuric acid
Solids concentration 20-25 grams in 3 litre
Frothing agent 1.5 mls of 5% W/v in 3 litre.

All flotation tests were performed using these conditions the only variables being the type and quantity of asbestos mixed with the talc.

Protected Document--Subject to Protective Order

Protected Document--Subject to Protective Order

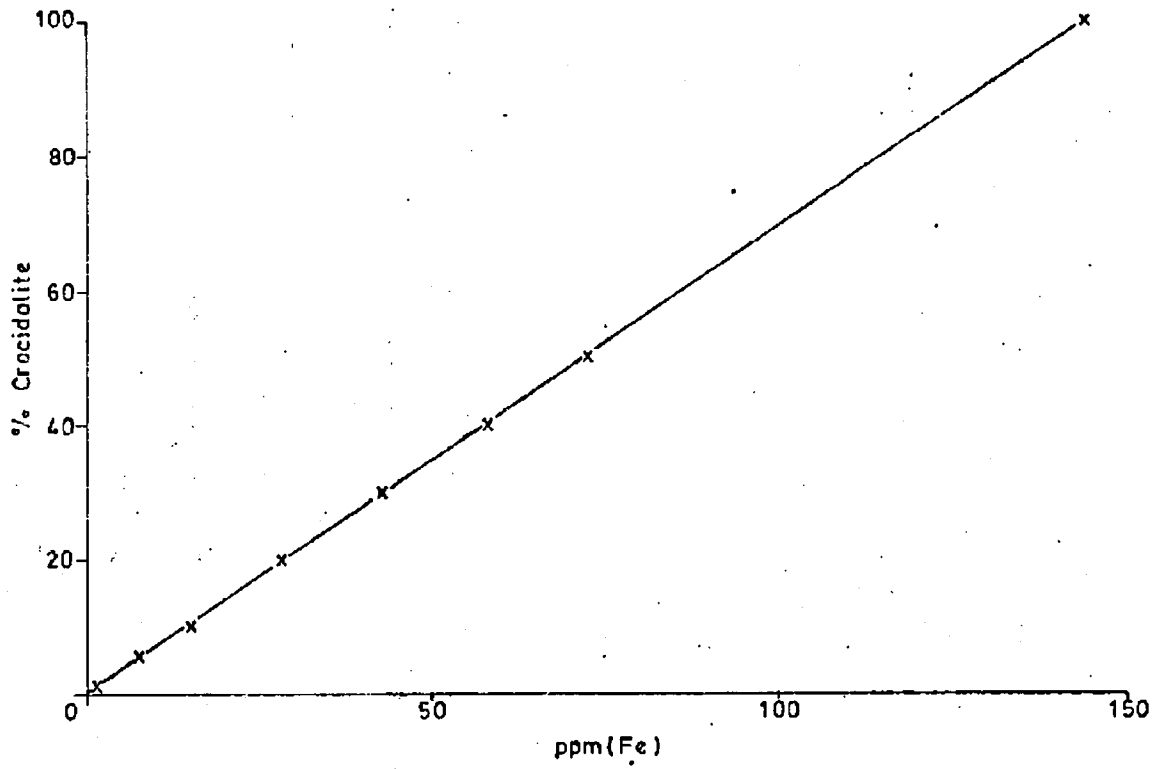


FIG. 2 CALIBRATION CURVE FOR CROCIDOLITE

JNJAZ55_000007962

To measure the separation of asbestos from talc a combination of wet chemical and X-ray diffraction techniques were used. For measuring crocidolite the iron concentration in the floated talc and also in the tailings was measured and the results compared with a standard curve prepared from the iron analyses of crocidolite/talc mixtures. Chrysotile was measured wet chemically by measuring the magnesium level obtained by digestion in concentrated hydrochloric acid and comparing the results with a standard curve prepared from the magnesium analyses of chrysotile/talc mixtures. The analysis technique for both iron and magnesium were as follows.

a) Wet Chemical determination of Crocidolite in Talc.

A small amount (0.05 - 0.1 gram) of the sample was fused in a P.T.F.E. crucible at approximately 350°C with 2 mls of (1:1) KOH and water. The fused pellet was then disssolved in 3 mls of concentrated H Cl. The solution was made up to a known volume and assayed for iron using an E.E.L. 240 atomic adsorption spectrophotometer. The amount of crocidolite was obtained by checking against a standard calibration curve as shown in Fig. 2.

b) Wet Chemical determination of Chrysotile in Talc.

Samples were dispersed in concentrated HCl (talc is not affected by HCl) and heated till dry. The samples were then retreated with dilute HCl. The resulting solutions were made up to a known volume and assayed for magnesium using an A.A.S. From standard calibration

Protected Document--Subject to Protective Order

Protected Document--Subject to Protective Order

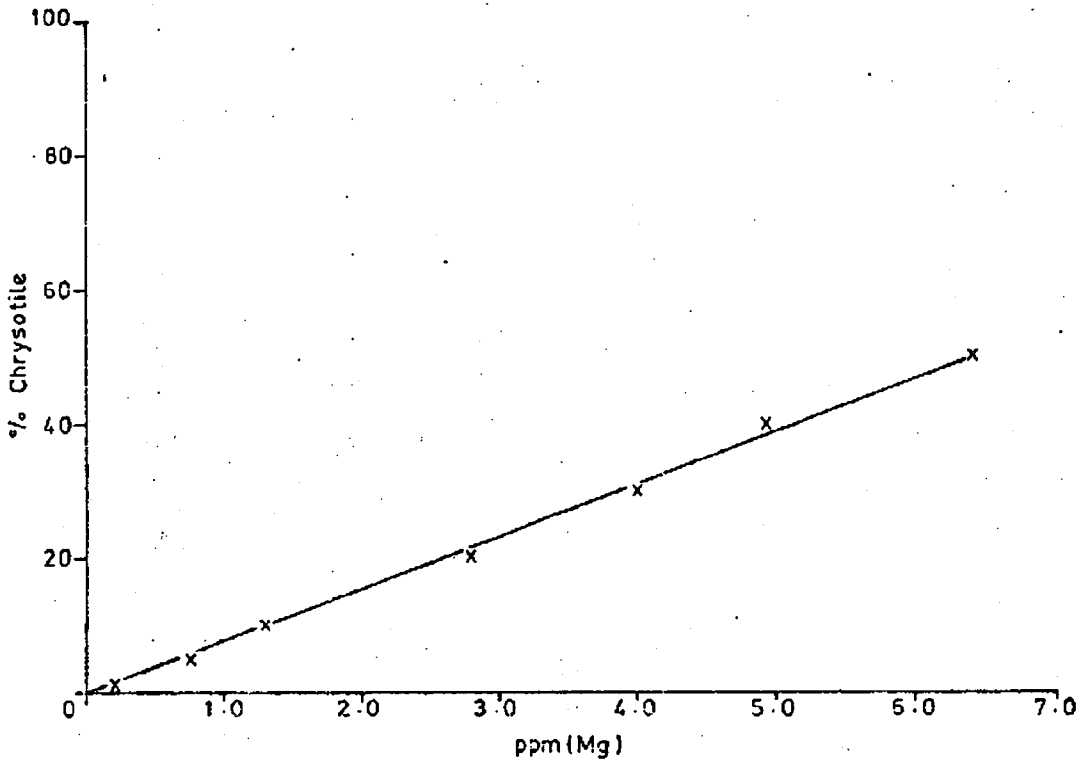


FIG. 3 CALIBRATION CURVE FOR CHRYSOTILE

JNJAZ55_000007964

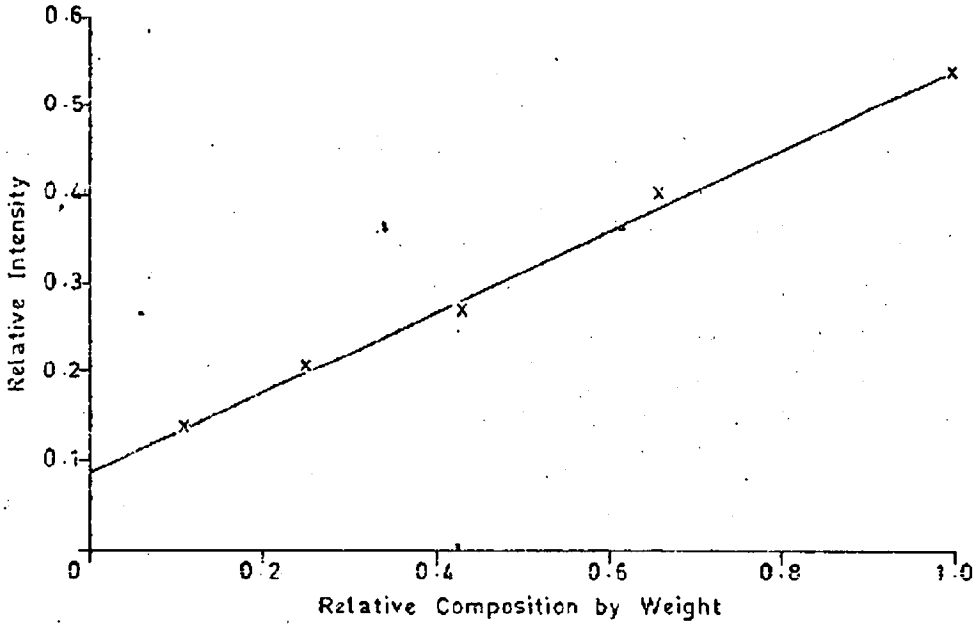


FIG. 4 DIFFRACTOMETER CALIBRATION CURVE FOR CROCIDOLITE IN TALC.

Protected Document--Subject to Protective Order

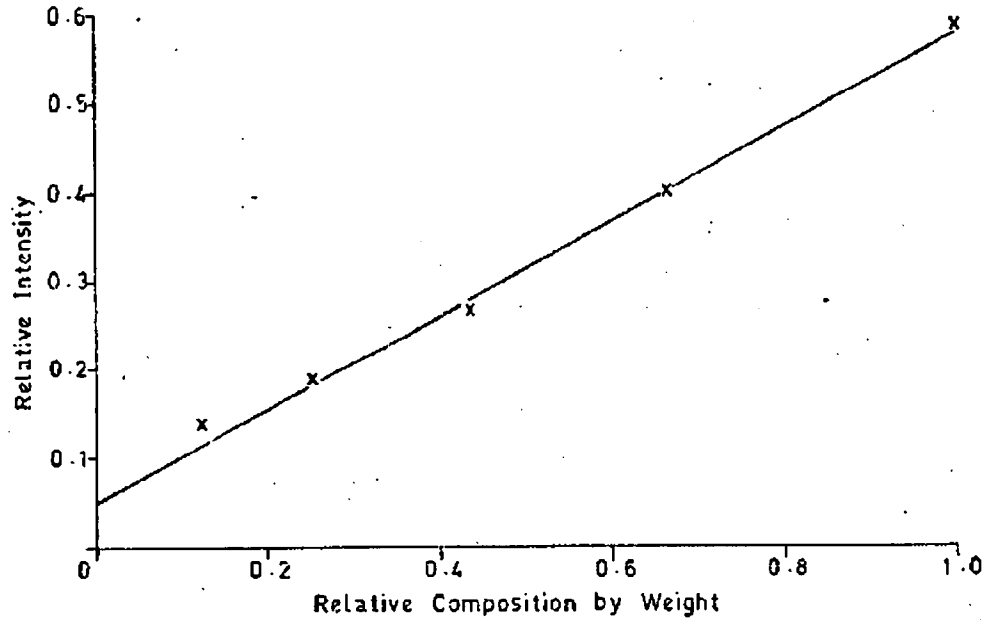


FIG. 5 DIFFRACTOMETER CALIBRATION CURVE FOR CHRYSOTILE IN TALC.

Protected Document--Subject to Protective Order

curves the amount of chrysotile was determined after correction for acid soluble magnesium in the talc tailings due to the presence of carbonate minerals. The carbonate magnesium was obtained by running flotation tests with no asbestos present and assaying the resultant tailings. The magnesium calibration curve is shown in Fig. 3.

X-ray determination of Chrysotile and Crocidolite.

The tailings samples were also subjected to X-ray diffraction measurements using a Siemens X-ray diffractometer. Quantitative readings were obtained by measuring the peak intensity of the amphibole reflection at 10.5 degrees 2θ and the chrysotile reflection at 12.2 degrees 2θ and expressing these as a ratio with the main talc reflection at approximately 9.0 degrees 2θ. This ratio figure was then compared with a calibration curve constructed from peak ratio figures obtained from mixtures of talc and the various asbestos samples these are shown in Figs. 4 and 5. The use of a ratio of chrysotile to talc peak and crocidolite to talc peak removed the necessity of using an internal standard for quantification purposes.

Care was taken in the preparation of X-ray standards to ensure good mixing and this was performed in aqueous suspension using the froth flotation cell as a mixer. Xlray powder preparations were investigated also and both pressed and loose specimens were examined. The loose powder preparation was preferred and used in all the measurements. Copper radiation was used throughout the

Protected Document--Subject to Protective Order

and 41% for the chrysotile in the same tailings fractions.

The results show that quantity of asbestos in the tailings increases dramatically with increase in asbestos content of the original asbestos/talc mixture. Also the fact that two asbestos minerals may be contained in the same sample has very little effect upon the recovery of each mineral in the tailings. The highest concentration ratios (K) obtained in these tests were with the 1% asbestos/talc mixtures where the asbestos was concentrated in the tailings from 1% to 35% approximately.

The tailings fractions were all subjected to X-ray diffraction examination as well as wet chemical analysis and the comparison of some of the quantitative results obtained are given in Table 4. Figs.10-12 illustrate portions of diffractometer traces obtained from separated tailings fractions of tests performed upon 1% crocidolite, 1% chrysotile, 1% crocidolite and chrysotile in talc mixtures.

Tests with 0.5% additions of actinolite in talc were also performed and Fig.13 illustrates the diffractometer trace obtained from a single stage separation of actinolite from talc. No quantification was performed for this sample as there was insufficient actinolite to prepare diffraction standards.

Protected Document--Subject to Protective Order

Conclusions.

Froth flotation would appear to be a most efficient means of separating amphibole and serpentine asbestos from talc for both detection and quantification of the asbestos phase. No interference appears to occur when two asbestos minerals are present and are being separated. Two stage separation is slightly more efficient than a single stage separation.

The most suitable flotation conditions for separation were found to be pH - 2.0 with a solids concentration of 6 - 8 grams per litre of water in the flotation cell. The most suitable frothing agent was found to be Frother 65 produced by Cyanimide Ltd.

Protected Document--Subject to Protective Order

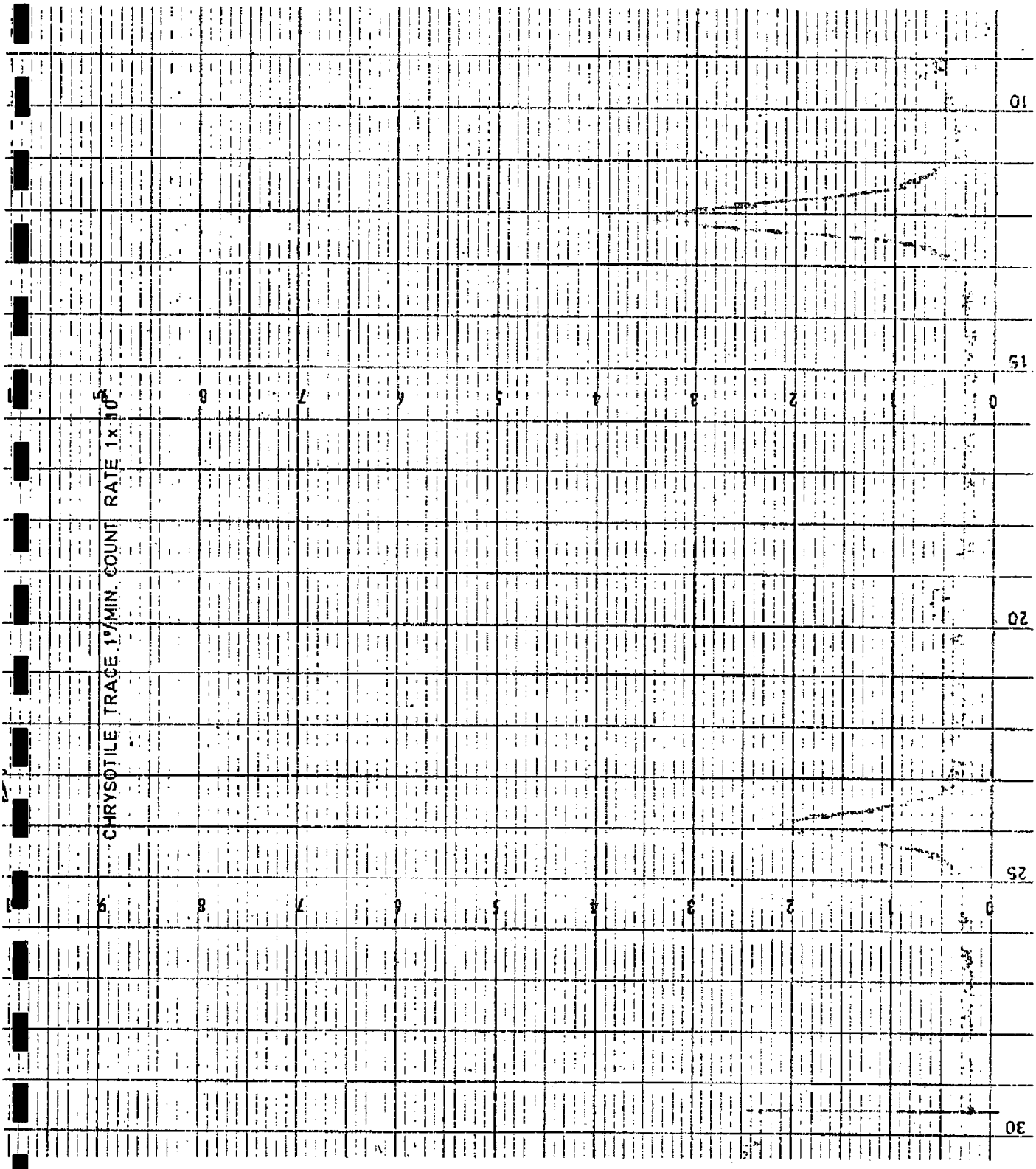


FIGURE 6

Protected Document--Subject to Protective Order

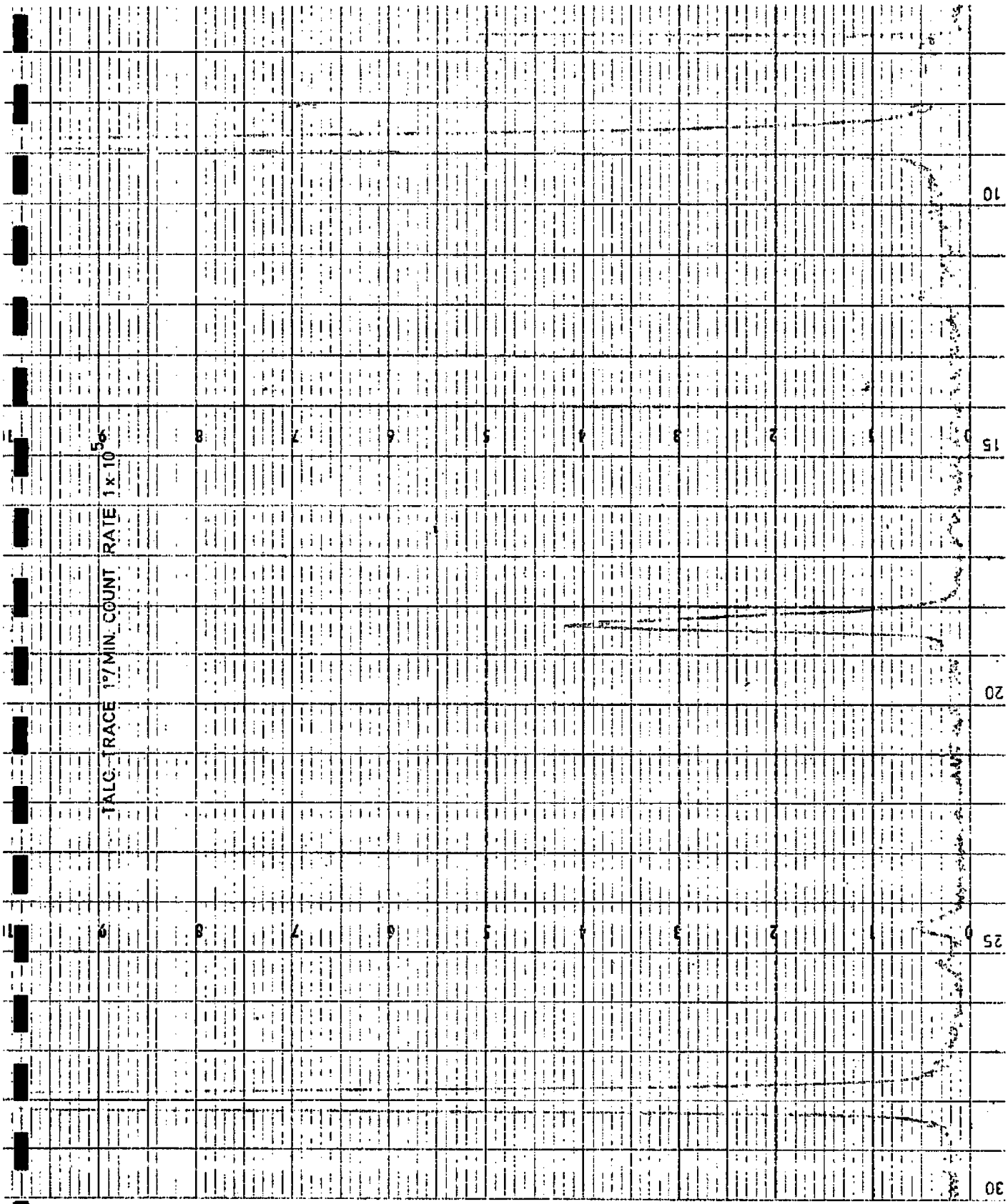


FIGURE 7

Protected Document--Subject to Protective Order

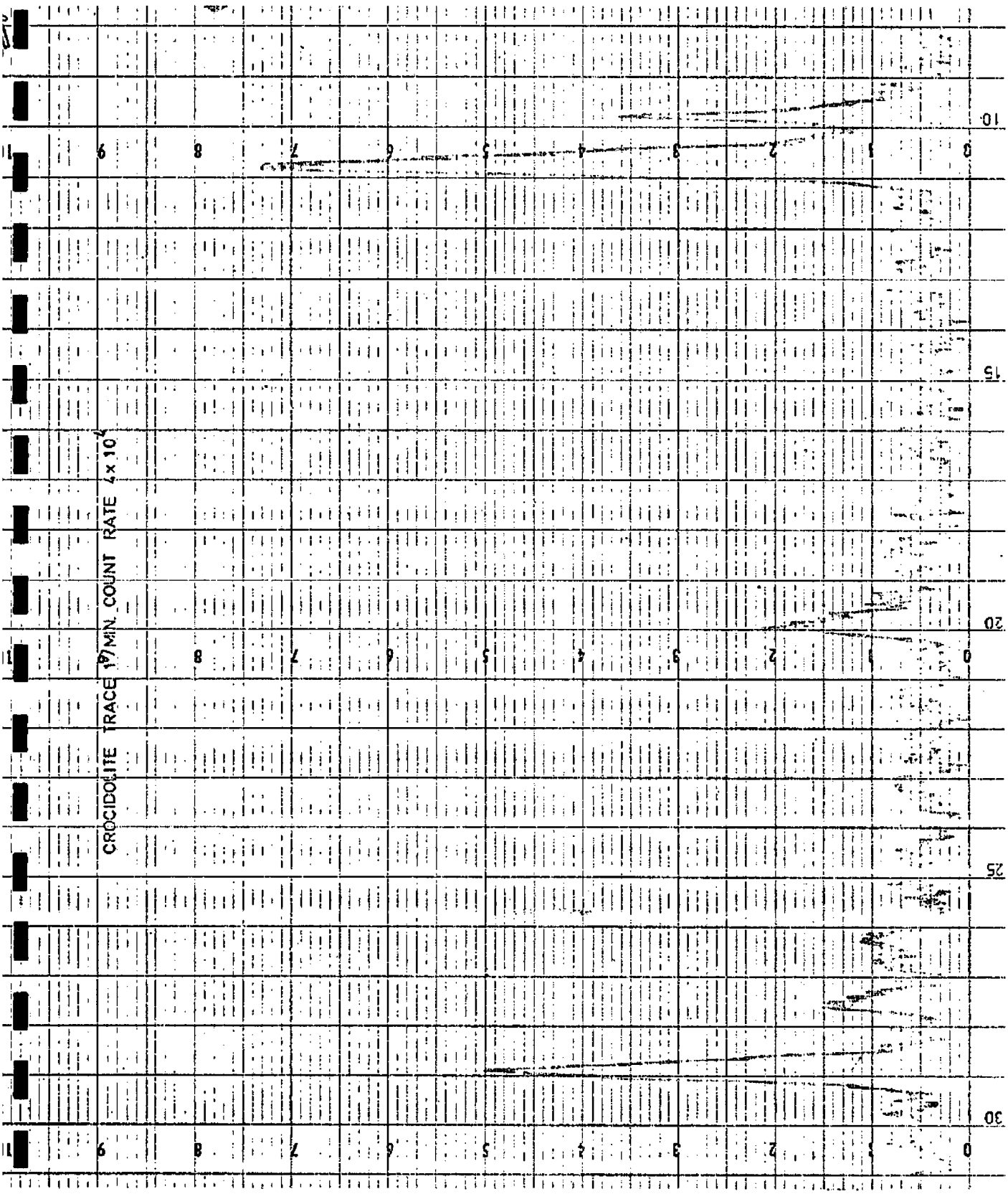


FIGURE 8

Protected Document--Subject to Protective Order

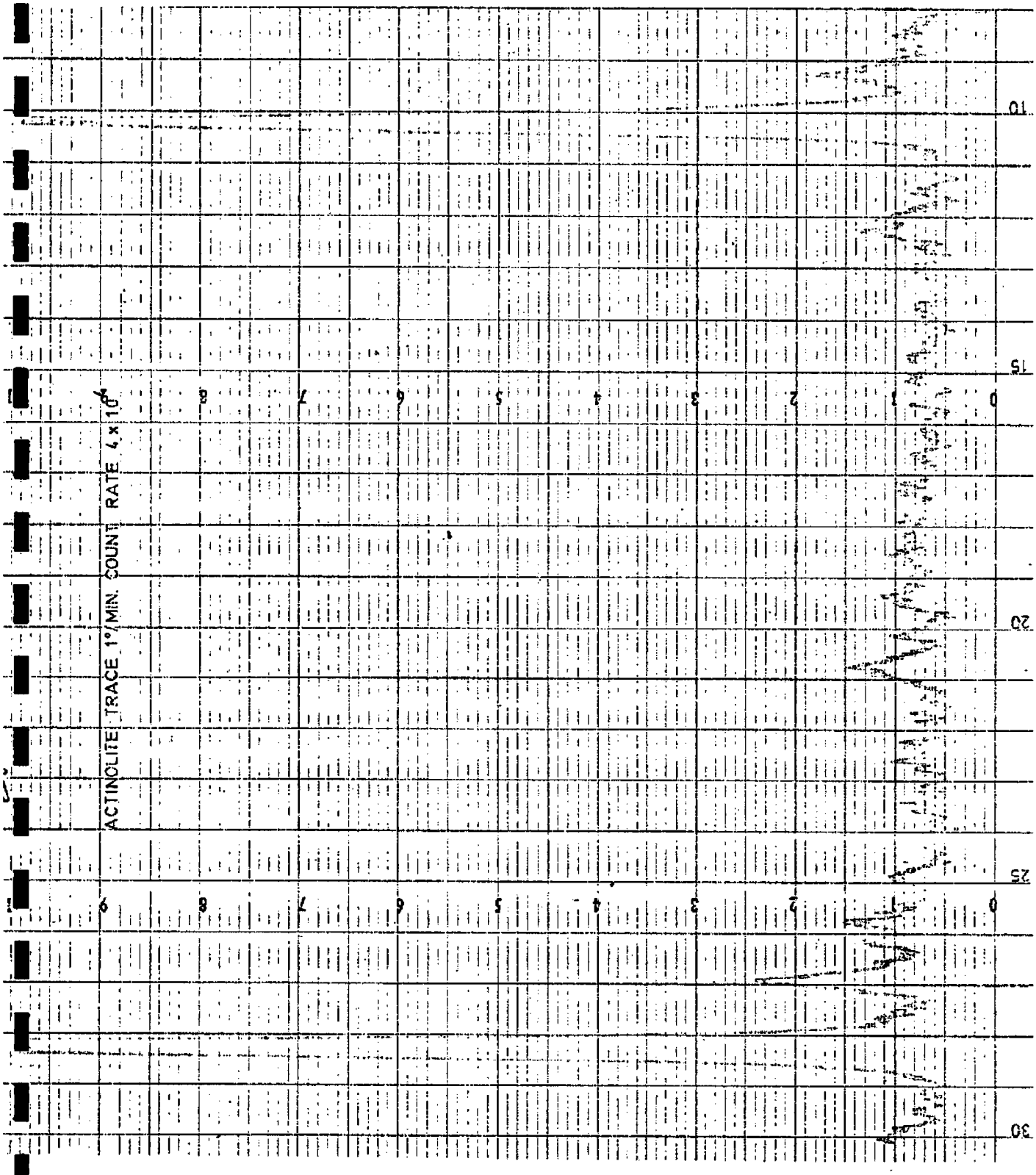


FIGURE 9

Protected Document--Subject to Protective Order

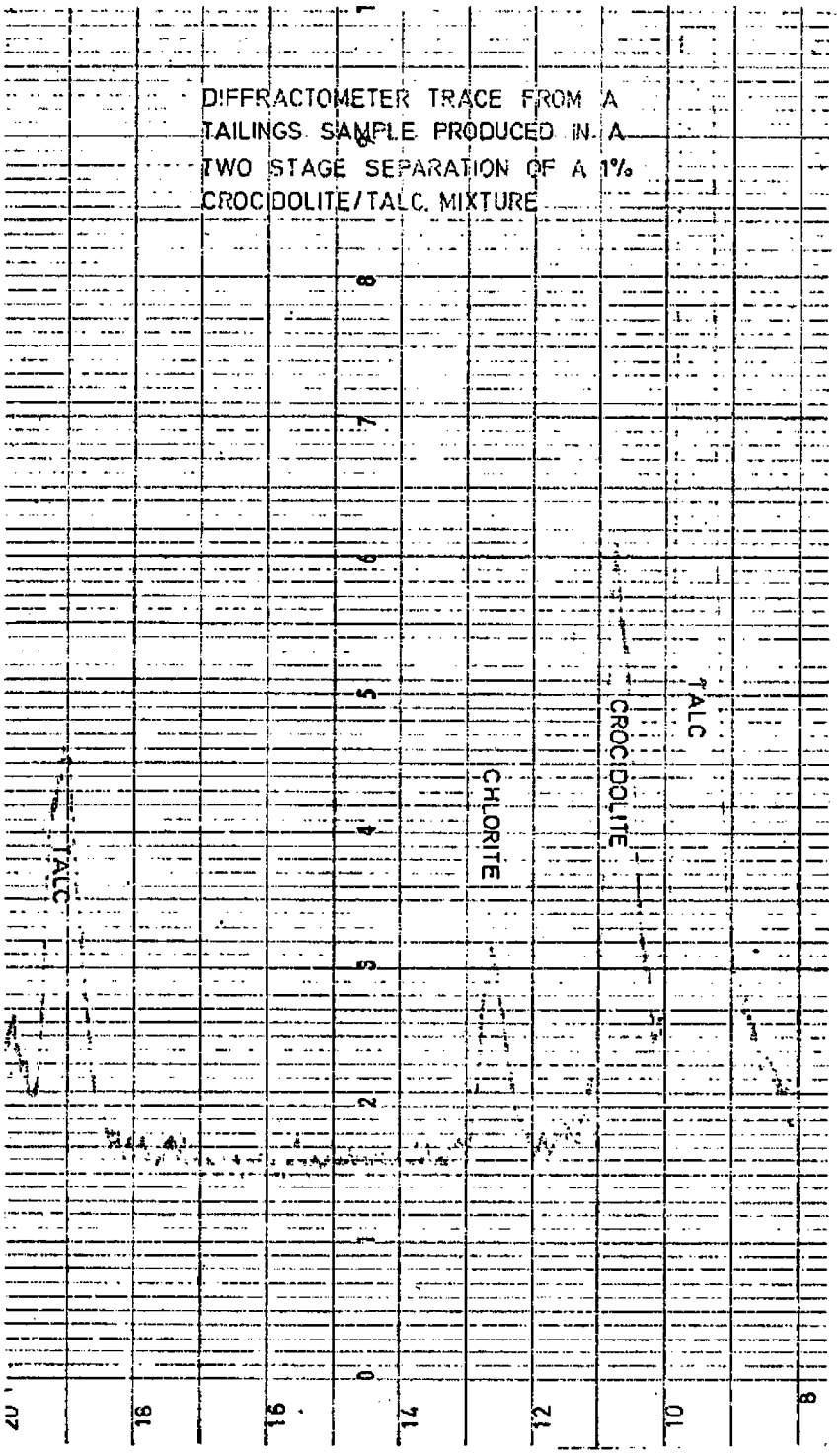


FIGURE 10

Protected Document--Subject to Protective Order

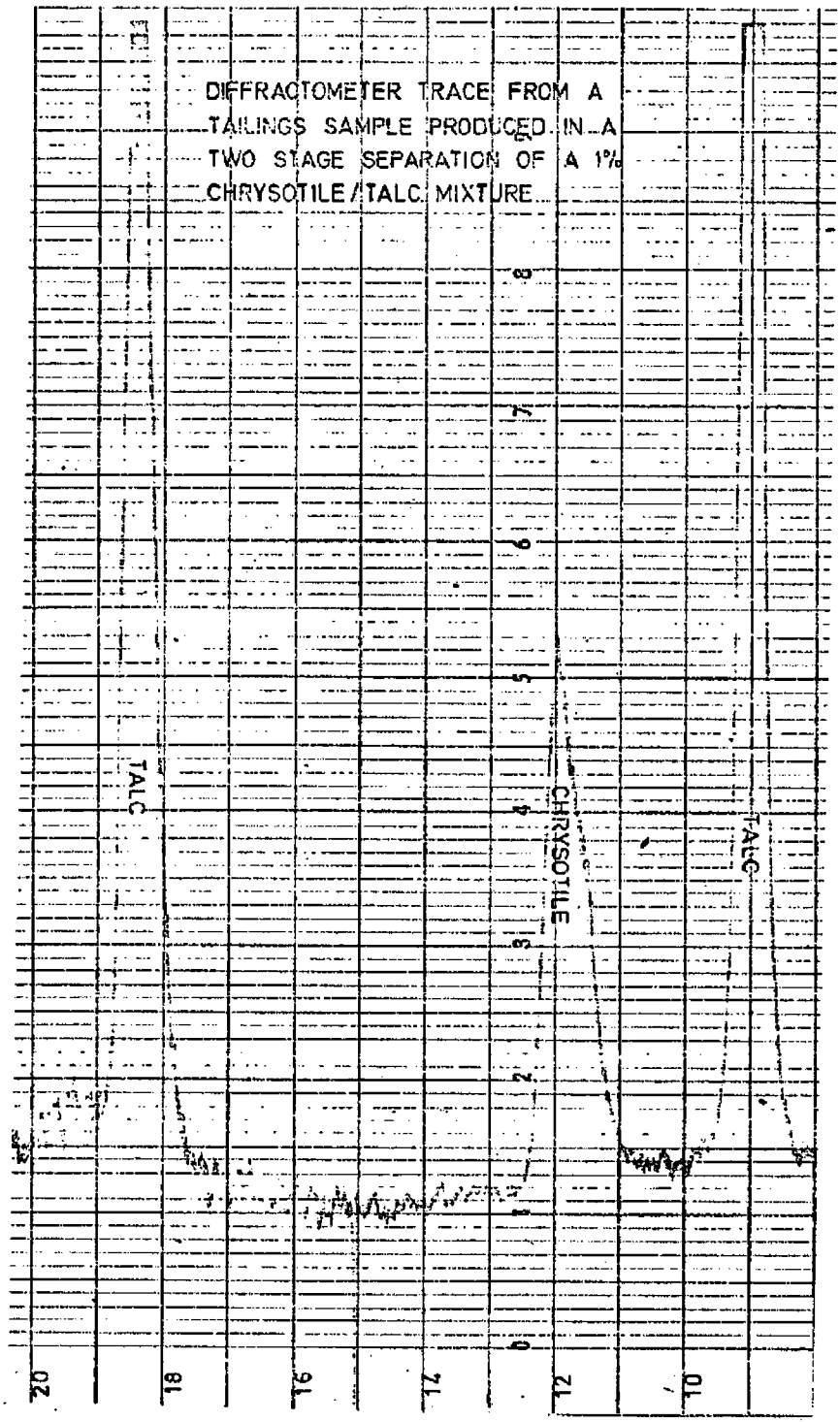


FIGURE 11

Protected Document--Subject to Protective Order

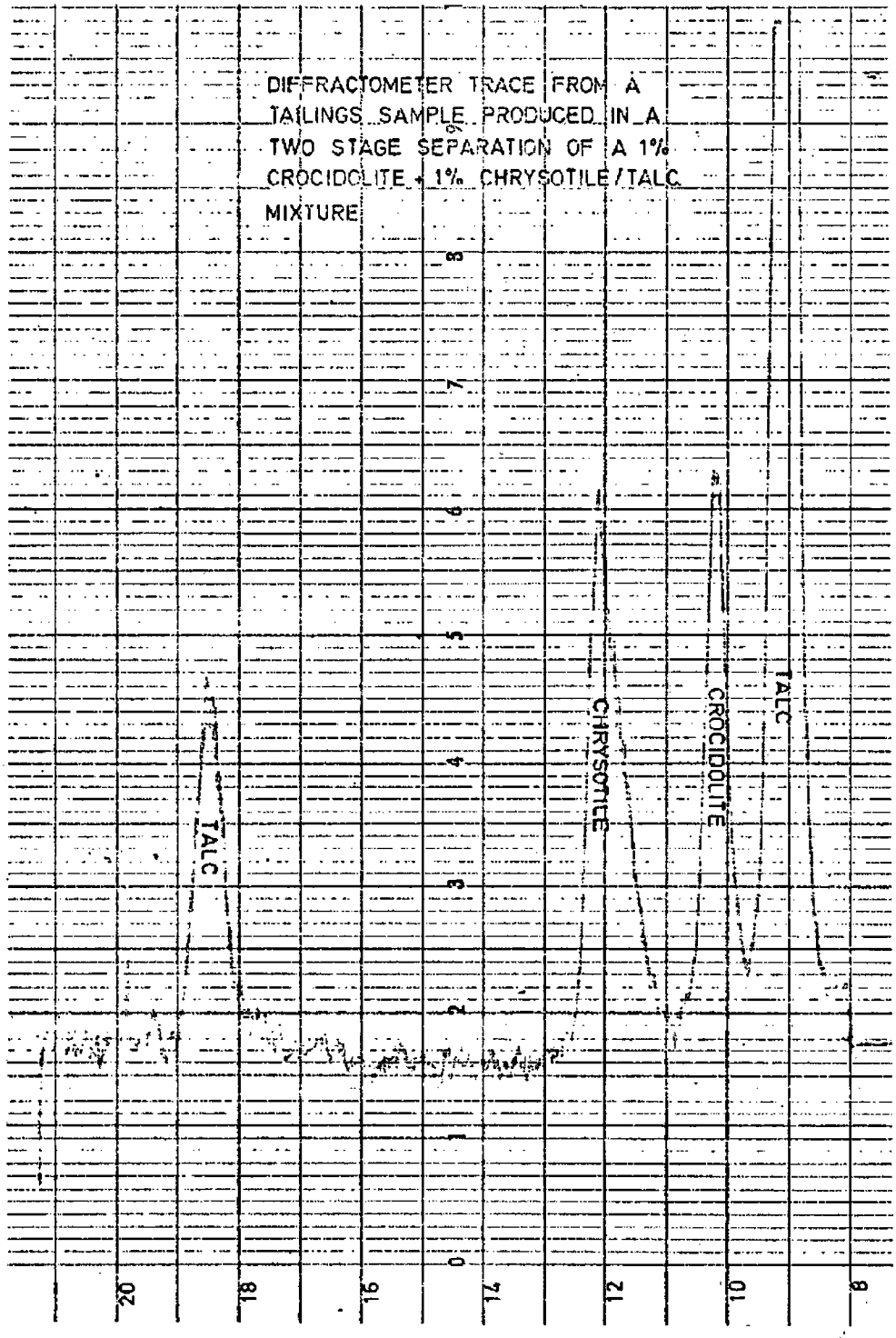


FIGURE 12

Protected Document--Subject to Protective Order

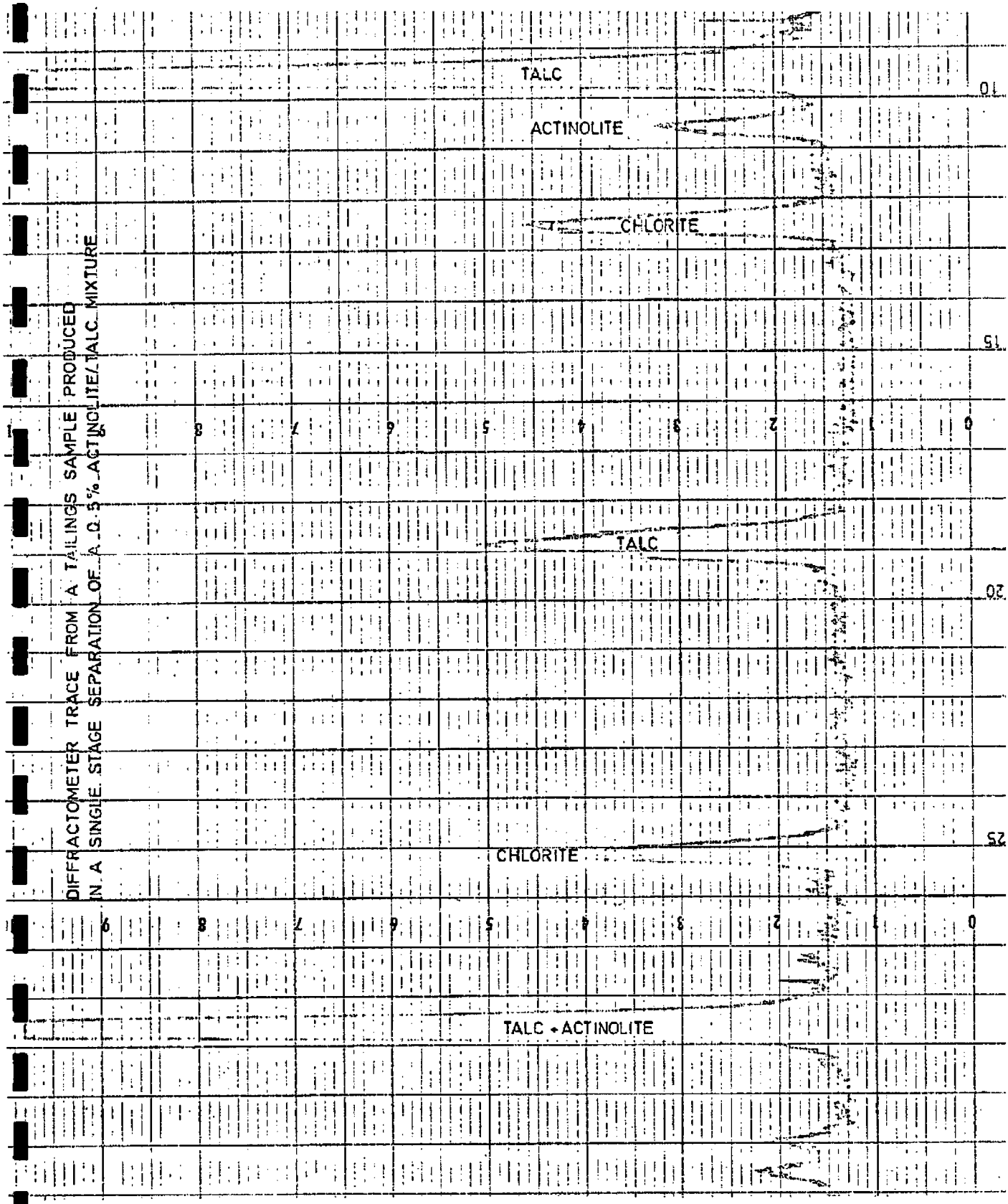


FIGURE 13

Protected Document--Subject to Protective Order