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Synthesis of *Cis-* and *Trans-* 1,3,5,7-Tetranitro-1,3,5,7- Tetraazadecalin, Two New Energetic Materials

by
Rodney L. Willer
Research Department

JANUARY 1983

NAVAL WEAPONS CENTER
CHINA LAKE, CALIFORNIA 93555



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FOREWORD

The Navy has a need for new energetic compounds which are both dense and energetic and which also have high thermal stability and low sensitivity to impact. These compounds could be used as ingredients in new propellant and explosive formulations which would simultaneously optimize both the performance and safety of new weapons systems. This report describes the synthesis and preliminary characterization of two such compounds, *cis*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin and *trans*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin.

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The work was reviewed for technical accuracy by R. L. Atkins and A. T. Nielsen.

Approved by
E. B. Royce, *Head*
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(U) The synthesis and preliminary characterization of two new energetic materials, *cis-* and *trans-*1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin are described.



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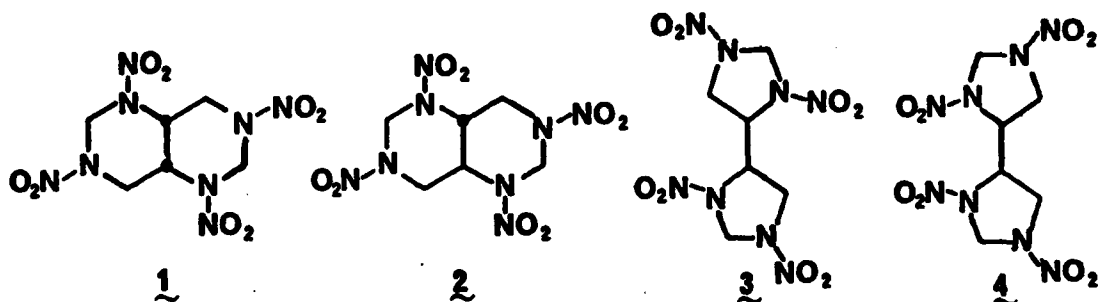
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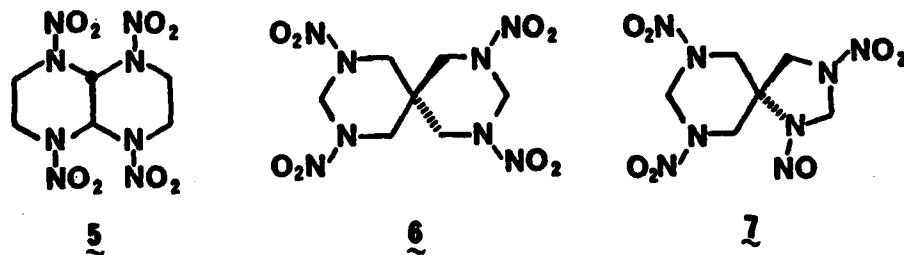
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INTRODUCTION

This paper describes the synthesis and preliminary characterization of two new tetranitramino compounds, *cis*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin, **1**, and *trans*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin, **2**. Also isolated and partially characterized were two by-products of the synthesis of **1** and **2**, *d,l*-4,4'-bi-(1,3-dinitro-1,3-diazacyclopentane), **3**, and *meso*-4,4'-bi-(1,3-dinitro-1,3-diazacyclopentane), **4**. This work is a continuation of our work on developing new methodology for the



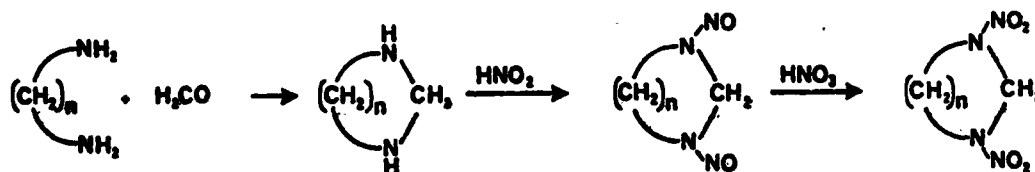
synthesis of polynitramino compounds and establishing the effect of stereochemistry and isomerization on the physical and chemical properties of polynitramino compounds.



Our previous work in this area has resulted in the synthesis of *trans*-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin, $\tilde{5}$,¹ 2,4,8,10-tetranitro-2,4,8,10-tetraazaspiro[5.5]undecane, $\tilde{6}$,² and an improved synthesis of 1,3,7,9-tetranitro-1,3,7,9-tetraazaspiro[4.5]decane, $\tilde{7}$.³ Compounds $\tilde{5}$ and $\tilde{7}$ are isomeric (i.e., both $C_6H_{10}N_8O_8$) yet they have very different densities (1.80 g/cc for $\tilde{5}$ ¹ and 1.70 g/cc for $\tilde{7}$ ³). This large difference in densities for $\tilde{5}$ and $\tilde{7}$ prompted us to formulate other structures which are isomeric with $\tilde{5}$ and $\tilde{7}$. Two such compounds were $\tilde{1}$ and $\tilde{2}$. We felt that these compounds were very attractive synthetic targets because they retained the basic decalin ring structure of $\tilde{5}$ but moved the nitramino substituents around the ring. This would allow us to determine the effects of placement of the nitramino further away from each other. Since we hoped to be able to synthesize both stereo isomers (*cis*- and *trans*-), we could determine the effect of the stereochemistry at the ring junction on the density of these polynitramino compounds.

Our strategy for the synthesis of $\tilde{1}$ and $\tilde{2}$ was based on our previously developed methodology for the synthesis of polynitramino compounds. This technique involves the trapping of an *in situ* generated 1,3-diazacycloalkane with nitrous acid to give a 1,3-dinitroso-1,3-diazacycloalkane followed by nitrolysis of the dinitroso compound to the corresponding 1,3-dinitro-1,3-diazacycloalkane with 100% nitric acid (see Scheme 1).² A retro-synthetic analysis of $\tilde{1}$ and $\tilde{2}$ based upon

SCHEME 1. Synthetic Methodology for 1,3-Dinitro-1,3-diazacycloalkanes.

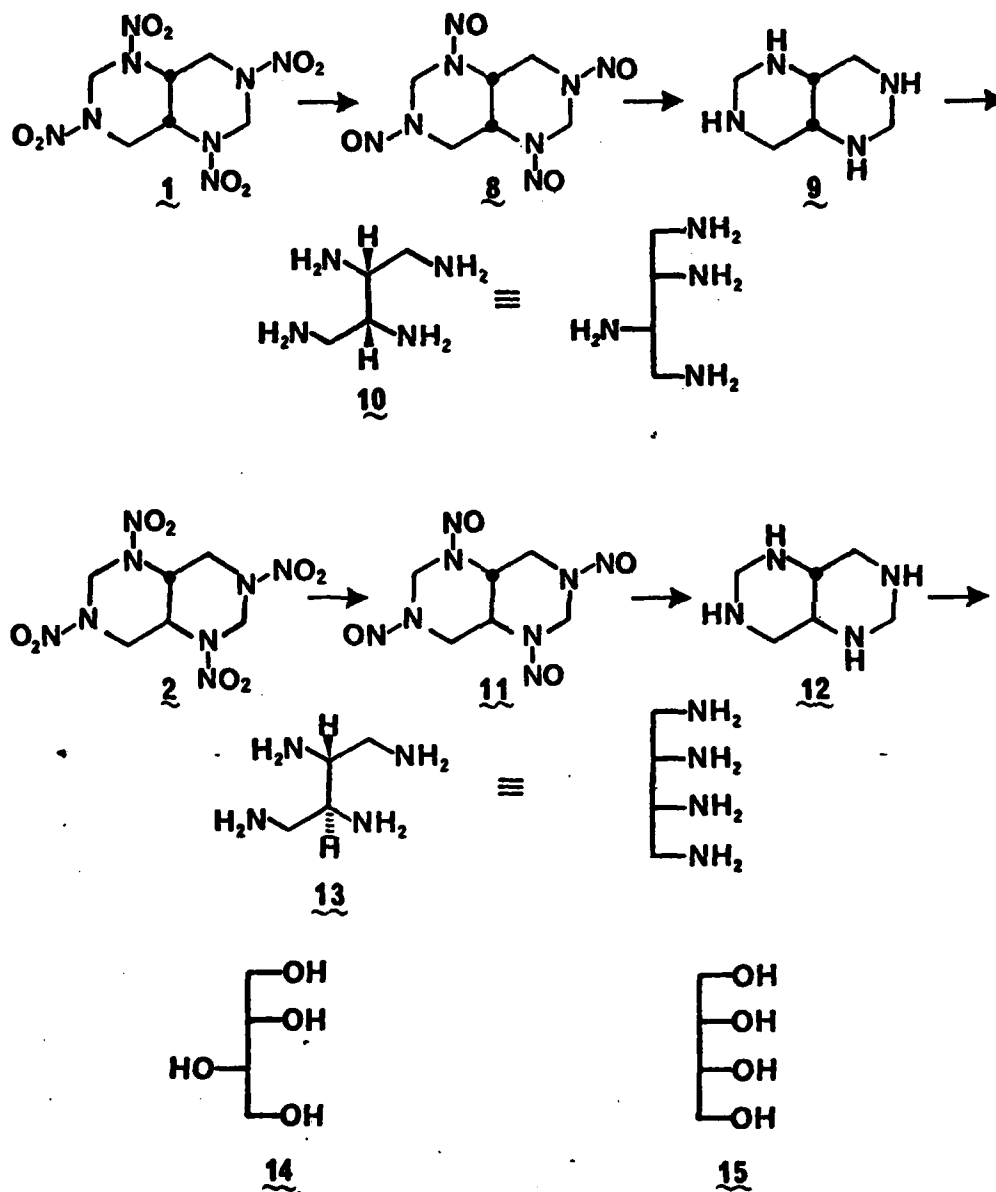


¹ Naval Weapons Center. *Synthesis of a New Explosive Compound, Trans-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin*, by R. L. Willer. China Lake, Calif., NWC, August 1981. 16 pp. (NWC TP 6303, publication UNCLASSIFIED.)

² ----- *Synthesis and Characterization of a New Insensitive High Energy Polynitramino Compound, 2,4,8,10-Tetranitro-2,4,8,10-tetraazaspiro[5.5]undecane (TNSU)*, by R. L. Willer, China Lake, Calif., NWC, March 1982. 10 pp. (NWC TP 6353, publication UNCLASSIFIED.)

³ ----- *Synthesis and Characterization of 1,3,5,7-Tetranitro-1,3,5,7-tetraazaspiro[4.5]decane (TNSD)*, by R. L. Willer, China Lake, Calif., NWC, January 1982. 10 pp. (NWC TM 4703, publication UNCLASSIFIED.)

this synthetic methodology indicated (see Scheme 2) that the required starting materials were *threo*-1,2,3,4-tetraaminobutane, 10, for 1 and *erythro*-1,2,3,4-tetraaminobutane, 13, for 2. Our strategy for the synthesis of the unknown 10 and 13 was to start with the corresponding tetrahydroxybutanes (threitol, 14, and erythritol, 15). These would be

SCHEME 2. Retro-Synthetic Analysis of 1 and 2.

converted to the amines by standard procedures^{2,4a,4b} of converting them first to their tetrabzenesulfonate derivatives using benzenesulfonyl chloride in pyridine, then converting the tetrabzenesulfonates to the tetraazides using sodium azide in dimethyl formamide (DMF), and finally reduction of the tetraazide to the tetraamine by catalytic hydrogenation.

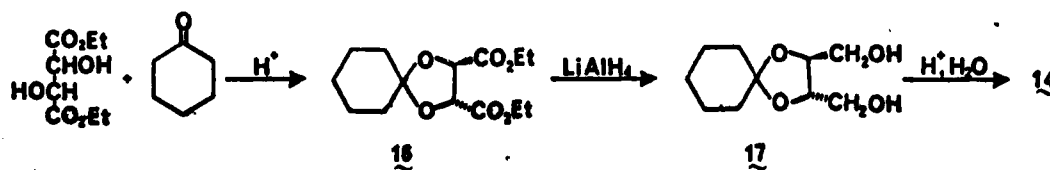
RESULTS

SYNTHESIS

Cis-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin (1)

The starting material, threitol (14), is commercially available, but quite expensive. We, therefore, chose to synthesize it from diethyl-L-tartrate using the procedure summarized in Scheme 3. This synthesis was adapted from one developed by R. U. Lemieux and J. Howard.⁵ Several minor modifications to the published procedures were made which both increased yields and simplified the procedures. These are detailed in the experimental section. An attempted direct reduction of diethyl-L-tartrate to L-threitol with LiAlH₄ failed.

SCHEME 3. Synthesis of L-threitol.



The conversion of the L-threitol to *threo*-1,2,3,4-tetraaminobutane, 10, is summarized in Scheme 4. The L-threitol was converted to its tetrabzenesulfonyl derivative, 18, in 70% yield using standard methodology. The tetrabzenesulfonate was then converted to the tetraazide,

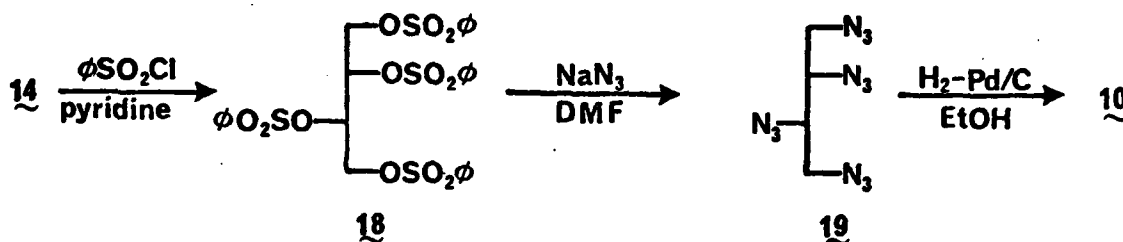
^{4a} E. B. Fleischer, et al. "Conversion of Aliphatic and Alicyclic Polyalcohols to the Corresponding Primary Polyamines," *J. Org. Chem.*, Vol. 36 (1971), p. 3042.

^{4b} P. Anselme. "Catalytic Reduction of 1,1,1-Tris/azidomethylethane to 1,1,1-Tris/aminemethylmethane," *Org. Prep. and Procedures*, Vol. 6 (1974), p. 103.

⁵ R. U. Lemieux and J. Howard. "The O-Inside Conformation of 1,3:2,4-Di-O-Methylene-L-Threitol," *Can. J. Chem.*, Vol. 41 (1963), p. 393.

19, using sodium azide in DMF at 100°C. The crude azide, 19, was then reduced to the *threo*-1,2,3,4-tetraaminobutane, 10, by catalytic hydrogenation in ethanol using 10% Pd/C as the catalyst. Since the reduction

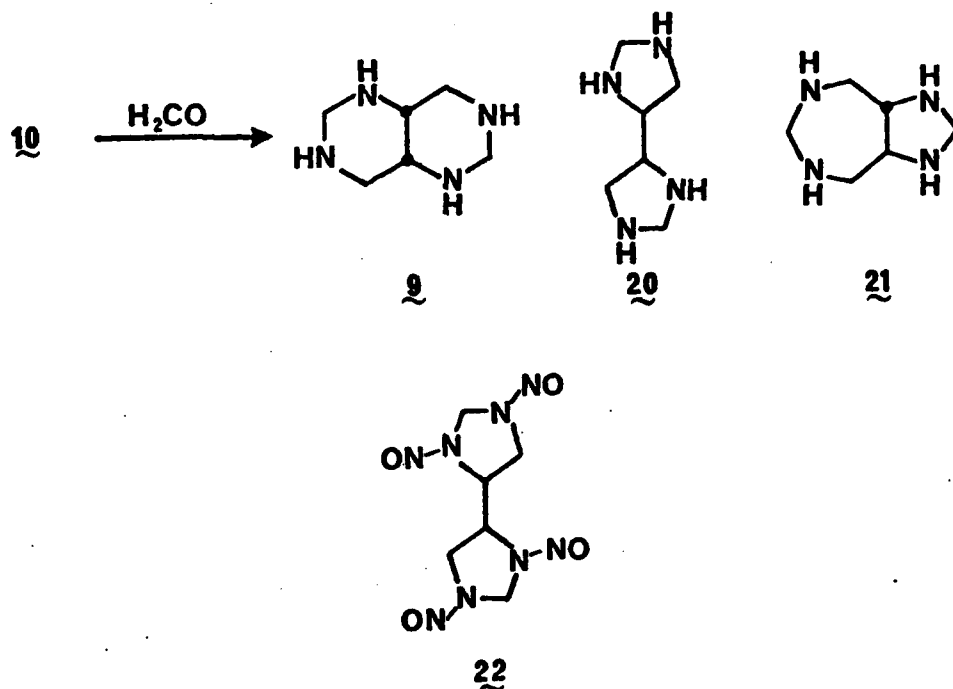
SCHEME 4. Synthesis of *Threo*-1,2,3,4-tetraaminobutane (10).



of a mole of azide requires one mole of hydrogen and releases one mole of nitrogen, no pressure drop is noted during the reduction. In order to introduce fresh hydrogen into the bottle, it is necessary to occasionally vent the bottle and refill it. The tetramine was characterized as its tetraacetamide derivative, 10b. Since the tetraamine was a hygroscopic liquid, it was difficult to get exact yield data. The best value for the combined yield of the displacement step and reduction step was 80-90%. The crude tetramine was used for the following synthetic procedures.

The best conditions for the formation of 9 from 10 were to dissolve 10 in a small amount of water and add the formaldehyde solution dropwise over a few minutes. This solution was stirred for an hour at 50°C. The solution was cooled to 5°C, and the required amount of sodium nitrite was added. After this had dissolved, the proper amount of 2M HCl was added in one portion. The tetranitroso product precipitated and was collected, washed, and dried. The yield for the product, based on starting tetrabenzenesulfonate, was 60-62%. The product was an amorphous powder and gave indications that it was a mixture of compounds. This is not unreasonable since there are three different ways for the *threo*-1,2,3,4-tetraaminobutane to cyclize, as summarized in Scheme 5. As shown from the results of the nitrolysis, the product is a mixture of the tetranitroso compounds, 8 and 22.

The nitrolysis of the product proceeded in rather poor yield to give a crude product which was clearly a mixture of two compounds by ¹H NMR spectroscopy. The major product was isolated in pure form by fractional recrystallization from acetone. This compound has been assigned the *cis*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin (1) structure based upon its ¹H NMR spectrum which showed an AB quartet for the isolated methylene groups (C_{2,6}) with a large chemical shift difference (≈1.9 ppm). This criterion has been used previously to assign structure in the analogous tetraxadecalin.⁵

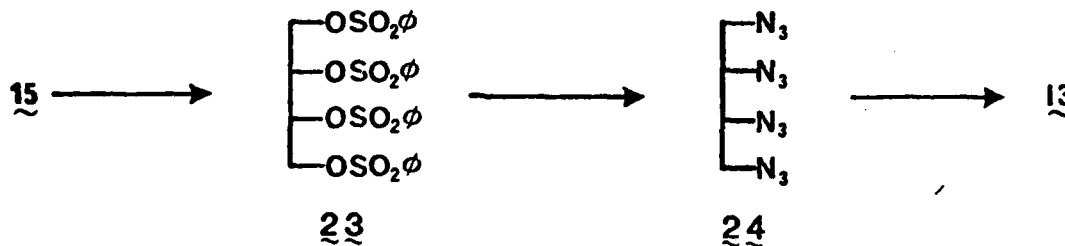
SCHEME 5. Possible Products From the Reactions of $\underline{10}$ With 2 Moles of Formaldehyde.

The minor product was isolated pure by preparative thin layer chromatography (TLC) of the mother liquors from the purification of $\underline{1}$. It was assigned the D-4,4'-bi-(1,3-dinitro-1,3-diazacyclopentane) structure, $\underline{3}$, on the basis of the small chemical shift difference for the protons of the isolated methylene groups (≈ 0.3 ppm).

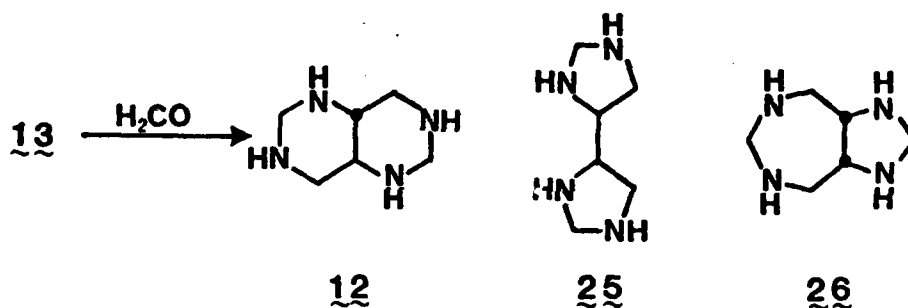
The ratio of $\underline{1}$ to $\underline{3}$ was observed to vary considerably depending on the conditions used for the reaction of the $\underline{10}$ with formaldehyde. When this reaction was run for a short time cold ($0-10^\circ\text{C}$), the proportion of $\underline{3}$ in the final product was increased quite substantially, even to the point where it was the major product. However, if the reaction was run as described above, the ratio of $\underline{1}$: $\underline{3}$ became approximately 10:1. This would seem to indicate that $\underline{20}$ is the kinetically favored product of the reaction of $\underline{10}$ with formaldehyde while $\underline{9}$ is the thermodynamically favored product.

Trans-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin (2)

In this case, the starting material, *meso*-erythritol, is commercially available at reasonable cost. The conversion of it to the desired *meso*-1,2,3,4-tetraaminobutane, $\underline{13}$, proceeded without complication through the tetrabenzenesulfonate, $\underline{23}$, and the tetraazide, $\underline{24}$, as outlined in Scheme 6. The details are given in the experimental section.

SCHEME 6. Synthesis of *Meso*-1,2,3,4-tetraaminobutane (13).

The reaction of the tetraamine, 13, with formaldehyde could again yield three products as summarized in Scheme 7. The reaction was run essentially as described for the *threo* isomer. This tetranitroso product

SCHEME 7. Possible Products from the Reaction of 9 with Formaldehyde.

was much better behaved. In fact, it could be recrystallized from DMF/H₂O and it gave a very nice IR spectrum. Nitrolysis of this product gave the crude *trans*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin, 2. The product could be recrystallized from DMF/H₂O to yield pure 2. Again, the assignment of structure was based upon the large (1.5 ppm) chemical shift difference for the protons of the isolated methylene groups (C_{2,6}). From the mother liquors of the recrystallization of 2, another product was isolated by preparative TLC. Small amounts of this product could also be obtained by further diluting the quench liquid and chilling overnight. This product was assigned the *meso*-4,4'-bi-(1,3-dinitro-1,3-diazacyclopentane) structure, 4, on the basis of the small shift difference (0.3 ppm) between the protons of the isolated methylene groups. The ratio of 2 to 4 can be estimated to be 20:1 from the ¹H NMR of the crude 2.

Physical and Chemical Properties of 1, 2, 3, and 4

The physical and chemical properties of compounds 1, 2, 3, and 4 that have been determined are summarized in Table 1.

TABLE 1. Physical and Chemical Properties
of 1, 2, 3, and 4.

	mp (°C)	Heat of formation Kcal/mole	Impact sensitivity (2.5 kg wt)	Density (g/cc)
1	236-237	+19.4	16.4	1.78
2	251-252	+26.5	65.0	1.75
3	213-214	+33.0	...	1.64
4	198-199

DISCUSSION

SYNTHESIS

The successful synthesis of 1 and 2 clearly demonstrates that the methodology which we had developed previously for the synthesis of 1,3-dinitro-1,3-diazacycloalkanes is applicable to more complex systems. We now plan to extend this methodology to even more complicated systems.

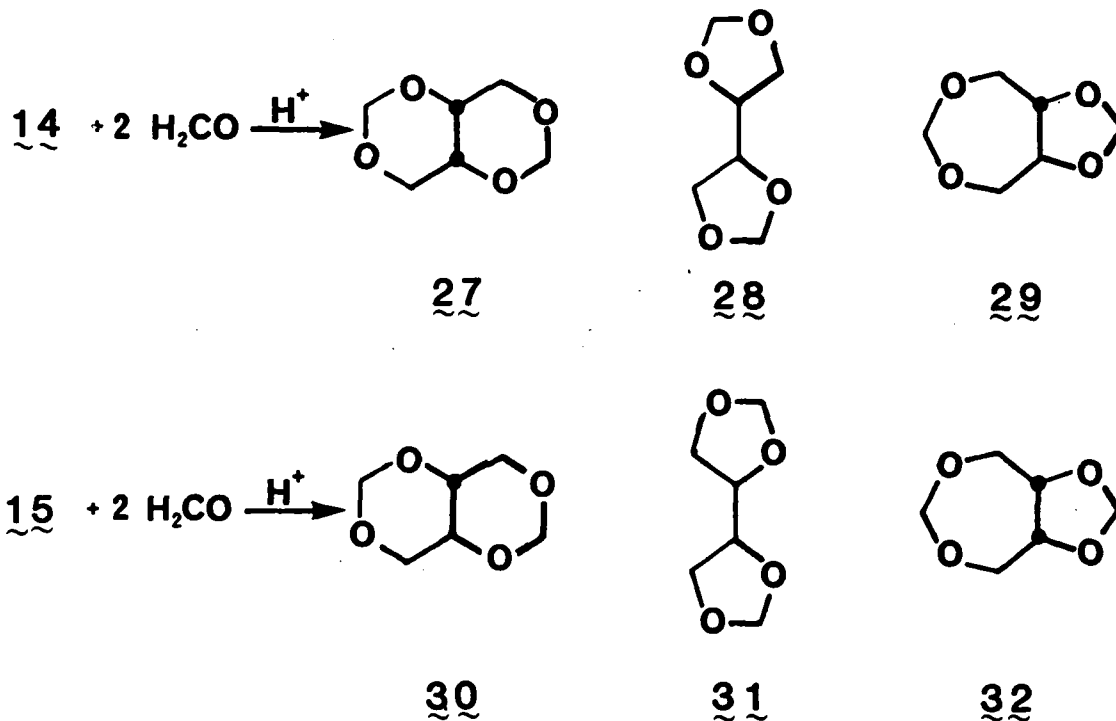
An interesting comparison can be made between this work and similar work on the reaction of threitol and erythritol with formaldehyde.⁵⁻⁷ As in this work there are three possible products from each reaction as summarized in Scheme 8. In the case of threitol, it has been found that the sole isolated product is the *cis*-1,3,5,7-tetraoxodecalin, 27.⁵ We observed that the reaction of *threo*-1,2,3,4-tetraaminobutane, 10, with formaldehyde gives a mixture of the tetraazadecalin, 9, and bi-(1,3-diazacyclopentane), 20, and that the ratio of 9:20 depends upon the

^{6a} I. J. Burden and J. Fraser Stoddart. "Isomerism in Bicyclic Diacetals, Part 1. 1,3:2,4- and 1,4:2,3-Di-O-Methylene Erythritol," *J. Chem. Soc., Perkins 1* (1975), pp. 666-674.

^{6b} I. J. Burden and J. Fraser Stoddart. "Constitutional Isomerism in Bicyclic Diacetals and the Conformational Behavior of *Cis*-Fused 1,3,6,8-Tetraoxabicyclo[5,3,0]decanes," *Chem. Comm.*, 1974, pp. 863-864.

⁷ R. B. Jensen, et al. "Reactions Between Formaldehyde and Polyhydric Alcohols, I. Five-, Six-, Seven-, and Twelve-Membered Cyclic Acetals from *Meso*-Erythritol and Formaldehyde," *Acta Chemica Scandinavica B29*, 1975, pp. 373-378.

SCHEME 8. Possible Products From the Reaction of Threitol and Erythritol with Formaldehyde.



reaction conditions. Since the reaction of threitol with formaldehyde was run under conditions which should favor the formation of the thermodynamic product (reflux, acid catalysis) these results compare favorably with ours run at higher temperatures where we obtained a 10:1 mixture of $\underline{9}$ and $\underline{20}$. The reaction of erythritol with formaldehyde has been found to give an $\approx 10:1$ mixture of the *trans*-1,3,5,7-tetraoxodecalin, $\underline{30}$, and the 1,3-dioxalano-1,3-dioxepane, $\underline{32}$;^{6,7} whereas we obtained a 20:1 mixture of the tetraazadecalin, $\underline{12}$, and the bi-(1,3-diazacyclopentane), $\underline{25}$, and none of the 5:7 fused compound $\underline{26}$. The reasons for these differences in the erythro cases are not clear.

PHYSICAL AND CHEMICAL PROPERTIES

Melting Points

As might be expected, all four compounds have relatively high melting points with both decalins ($\underline{1}$ and $\underline{2}$) having higher melting points than the bi-cyclopentanes ($\underline{3}$ and $\underline{4}$). Compound $\underline{2}$ has one of the highest melting points of the known polynitramines.

Densities

According to the Holden method,⁸ compounds 1-4 have the same predicted density of 1.74 g/cc. The measured densities vary considerably from this value (see Table 1). That 1 has a density greater than 2 is in line with the fact that *cis*-decalin, 33, has a higher density than *trans*-decalin, 34.⁹



d = 0.8967

33

d = 0.8700

34

That 3 should have such a low density is in accord with the observation that compounds of this type tend to have lower densities than the parent compounds because the rings are forced to be perpendicular to each other. The densities of some other bi-compounds and the parent compounds follow.¹⁰⁻¹² We have measured and included the density of 1,3-dinitro-1,3-diazacyclopentane, 38, the parent compound of 3.

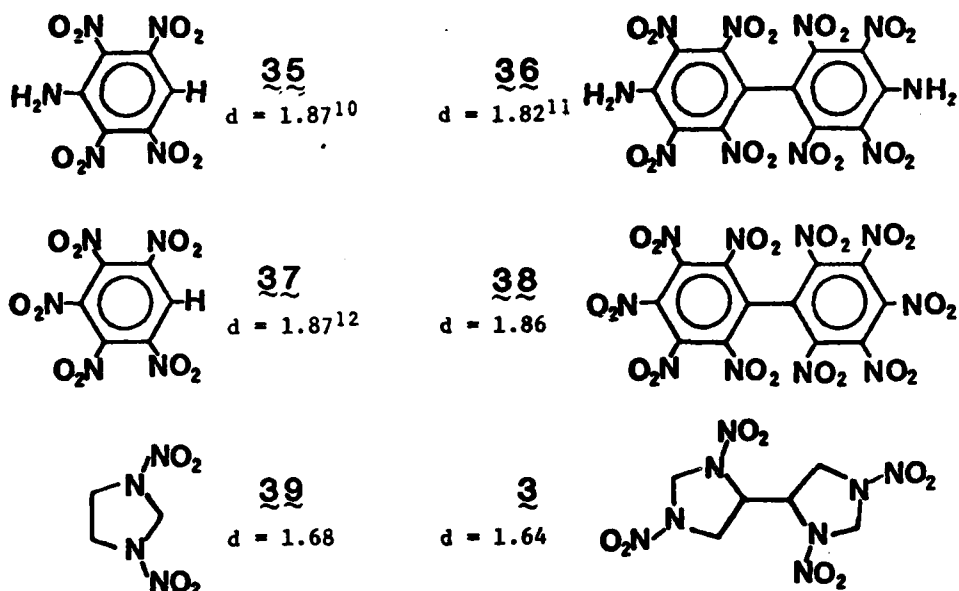
⁸ Naval Surface Weapons Center. *Estimation of Normal Densities of Explosive Compounds from Empirical Atomic Volumes*, by D. A. Cichra, J. R. Holden and C. Dickinson. Silver Spring, Md., NSWC, 1980. 39 pp. (NSWC Report TR79-273, publication UNCLASSIFIED.)

⁹ The Chemical Rubber Co. *Handbook of Chemistry and Physics*, 45th ed. Robert C. Weast, ed. Cleveland, Ohio, 1964, p. C281.

¹⁰ Lawrence Livermore National Laboratory. *LLNL Explosive Handbook. Properties of Chemical Explosives and Explosive Simulants*, by B. M. Dobratz. Livermore, Calif., LLNL, March 1981. (Report No. UCRL-52997, publication UNCLASSIFIED.)

¹¹ Naval Weapons Center. *Synthesis and Properties of a New High Explosive, 4,4'-Diamino-2,2',3,3',5,5',6,6'-octanitrobiphenyl (CL-12)*, A. T. Nielsen, et al. China Lake, Calif., NWC, April 1981. 22 pp. (NWC TP 6212, publication UNCLASSIFIED.)

¹² C. Coon, SRI International, (unpublished observations).

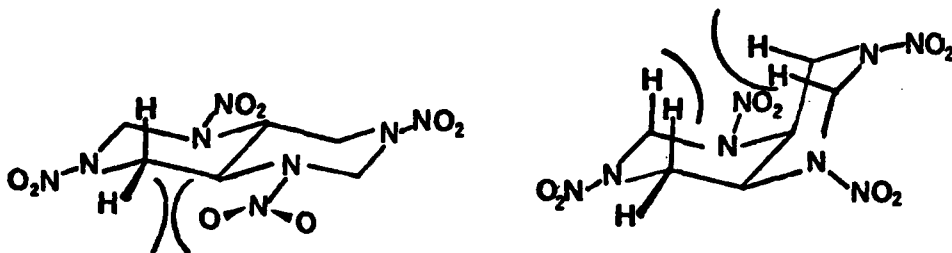


Impact Sensitivities

Impact sensitivities were measured only on compounds $\underline{\underline{1}}$ and $\underline{\underline{2}}$. The great difference (16 vs. 65 cm) in the values for $\underline{\underline{1}}$ and $\underline{\underline{2}}$ would seem to be unreasonable. However, it should be noted that there was a great difference in the crystal sizes of samples used for this measurement. The sample of compound $\underline{\underline{1}}$ we used was composed of large crystals ($\approx 1 \text{ mm}^2$) while the sample of $\underline{\underline{2}}$ was a microcrystalline material. Since several studies have noted a relationship between crystal size and impact sensitivity,¹¹ the large difference between the measured values for $\underline{\underline{1}}$ and $\underline{\underline{2}}$ may in part be simply a function of particle size and not a true measure of their respective inherent impact sensitivities.

Heats of Formation

The measured heats of formation of $\underline{\underline{1}}$, $\underline{\underline{2}}$, and $\underline{\underline{3}}$ seem to reflect the amount of steric strain present in the molecules as estimated by inspection of molecular models of the compounds. The *trans*-decalin, $\underline{\underline{2}}$, is highly strained because of a very unfavorable interaction between the 1,5 nitro groups and the 4,8 methylene groups. As shown below, this interaction is absent in the *cis*-decalin.



EXPERIMENTAL

Densities were determined on a Systems, Science, and Software type 6102 gas pycnometer and are the average of three separate determinations. ^1H NMR spectra were recorded on a Varian EM-360 or on an XL-100 NMR spectrometer. Impact sensitivities were measured on 35-mg samples on a Model 12 impact machine. Heats of combustions were determined on a Parr adiabatic bomb calorimeter. IR spectra were recorded on a PE 137 spectrometer. Elemental analyses were determined by Galbraith Laboratories, Knoxville, Tennessee.

DIETHYL-2,3-O-CYCLOHEXYLIDINE-L-TARTRATE (16)

A solution of diethyl tartrate (206.19 g, 1 mole) and cyclohexanone (147.2 g, 1.5 mole) dissolved in 600 ml of benzene was prepared. To this solution 5 g of *p*-toluene sulfonic acid monohydrate was added. The solution was refluxed and the water collected by means of a Dean-Stark trap. After the water had stopped collecting (16 hours) the solution was cooled, washed with 10% NaOH (100 ml), then dried over MgSO_4 . The solution was filtered and the solvent removed at reduced pressure. The crude product was vacuum distilled to give 175.6 g of product (0.61 mole, 61%) with a b.p. of 124–126°C at 0.15 mm (lit⁵ 142°C at 10 mm).

IR (See Appendix A, Figure A-1).

NMR (See Appendix B, Figure B-1).

L-THREITOL (14)

Diethyl-2,3-O-cyclohexylidene tartrate (57.2 g, 0.2 mole) was dissolved in 100 ml of dry tetrahydrofuran (THF). This solution was added dropwise over a 1-hour period to a well-stirred slurry of 14.0 g of

lithium tetrahydroaluminate and 400 ml of dry THF. The reaction mixture was stirred for 2 additional hours then the reaction was worked up by the careful addition of water (14 ml), 15% NaOH (14 ml), and more water (28 ml). The resulting slurry was filtered and the solid material reslurried with 200 ml hot THF and refiltered. The combined THF solutions were concentrated at reduced pressure to yield the crude 2,3-O-cyclohexylidene-L-threitol, 17. The IR and NMR spectra of the crude product indicated it was very pure (see Appendix A, Figure A-2 and Appendix B, Figure B-2). Water (200 ml) and 1 ml of concentrated hydrochloric acid was added to the crude 17. This mixture was heated and the cyclohexanone/water azeotrope distilled off over a 1-hour period. The solution was cooled and extracted with 100 ml diethyl ether. The aqueous phase was concentrated at reduced pressure to yield a thick oil which crystallized upon the addition of 200 proof alcohol. The alcohol was removed at reduced pressure to yield the crude L-threitol, 20.42 g (0.18 mole, 90%). A small sample was recrystallized from 95% EtOH to give needles with a m.p. of 86-87°C (lit⁵ 86-88°C).

IR (See Appendix A, Figure A-3).

NMR (See Appendix B, Figure B-3).

L-THREITOL-TETRABENZENESULFONATE (18)

L-threitol (12.2 g, 0.1 mole) was dissolved in 200 ml dry pyridine. This solution was cooled (salt-ice bath) and benzenesulfonyl chloride (75 ml, 0.6 mole) was added dropwise over a 1-hour period. The cooling bath was removed and the mixture was stirred at room temperature for 16 hours. The solution was poured onto a mixture of 300 g ice and 100 ml concentrated HCl. The product oiled out. The aqueous layer was decanted from the oil. The oil was dissolved in a hot mixture of methanol (300 ml) and acetone (50 ml). The volume of the solution was reduced to 200 ml and the product allowed to crystallize. The product was collected and dried. The yield was 48.24 g (0.07 mole, 70%) of material with a m.p. of 112-114°C.

Analysis calculated for $C_{28}H_{26}O_{12}S_4$: C, 49.26; H, 3.81.
Found: C, 49.18; H, 4.00.

IR (See Appendix A, Figure A-4).

NMR (See Appendix B, Figure B-4).

THREI -1,2,3,4-TETRAAZIDOBUTANE (19)

L-threitol tetrabenzenesulfonate (20.46 g, 0.03 mole), sodium azide (10 g, 0.15 mole) and dry DMF (200 ml) were placed in a 500 ml flask. This solution was stirred at 100°C for 4 hours then cooled to 10°C and

diluted with 250 ml H₂O. The H₂O/DMF solution was extracted with ether (4 x 100 ml) and the combined ether extracts bath extracted with water (2 x 50 ml). The ether solution was dried over MgSO₄, filtered, and the ether removed at reduced pressure to give the crude *threo*-1,2,3,4-tetraazidobutane. The products contain a fair amount of DMF so no yield data can be obtained. No attempt was made to purify the product because of the known hazards of polyazido compounds.

IR (See Appendix A, Figure A-5).

NMR (See Appendix B, Figure B-5).

THREO-1,2,3,4-TETRAAMINOBUTANE (10)

The crude tetraazide was dissolved in 120 ml, 95% ethanol in a 500 ml Parr bottle and 1 g of 10% Pd/C was added. This mixture was hydrogenated at 55 psi with the tank shut off. Every hour the bottle was vented and fresh hydrogen was introduced. After 4 hours, the solution was filtered and the solvent removed at reduced pressure to yield the crude *threo*-1,2,3,4-tetraaminobutane. A small portion of this reacted with acetic anhydride gave the tetraacetamide derivative with a m.p. of 242-244°C.

Analysis calculated for C₁₂H₂₂N₄O₄: C, 50.33; H, 7.74; N, 19.57.
Found: C, 49.94; H, 7.92; N, 19.17.

IR (See Appendix A, Figure A-6).

NMR (See Appendix B, Figure B-6).

CIS-1,3,5,7-TETRANITROSO-1,3,5,7-TETRAAZADECALIN (8) and
D-(4,4')-BI-(1,3-DINITROSO-1,3-DIAZACYCLOPENTANE) (22)

The crude 10 was dissolved in 30 ml of distilled water and 5.0 g of 37% aqueous formaldehyde solution was added dropwise over a 5-minute period. The mixture was stirred at 50°C for an hour. The solution was cooled to 5°C and sodium nitrite (8.4 g, 0.12 mole) was added. When the sodium nitrite had completely dissolved, 60 ml of 2N HCl was added in one portion. The product precipitated and was collected and washed well with water. After drying the product weighs 4.61-4.80 g (0.018-0.019 mole, 60-62%).

IR (See Appendix A, Figure A-7).

NMR (See Appendix B, Figure B-7).

CIS-1,3,5,7-TETRANITRO-1,3,5,7-TETRAAZADDECALIN (1) and
D-4,4'-(1,3-DINITRO-1,3-DIAZACYCLOPENTANE) (3)

The crude product from the previous step was ground into a fine powder. A 3 g portion (0.012 mole) was then added over a 10-minute period to 45 ml of well stirred 100% nitric acid which was maintained at -30°C by means of a dichloroethane dry ice slush. After the addition was complete, the dichloroethane bath was removed and replaced with an ice water bath. The solution was stirred at 0°C for 20 minutes then the ice water bath was removed and replaced with a 40°C hot water bath. The mixture was stirred at 40°C for 15 minutes then poured onto 200 g of ice. After the ice had melted the quench solution was diluted to 500 ml. The crude product was collected by vacuum filtration. A second crop could be collected by allowing the mother liquor to stand over night. The first crop of crude product weighed 2.26-2.46 g after drying. The second crop weighed 0.75 g after drying. The total yield of crude product is approximately 3.02 g (0.009 mole, 80%). Depending upon the conditions used for the reaction of 10 with formaldehyde, the ratio of 1:3 varies from 15:1 to $\approx 1:1$.

NMR (See Appendix B, Figures B-8 and B-9).

Separation of 1 and 3

Mixtures Rich in 1. The entire crude product (≈ 3.0 g) was dissolved in acetone and filtered to remove insoluble materials. The solution was concentrated to 15 ml and a seed crystal of pure 1 was added. The solution was allowed to slowly evaporate until the volume of liquid was ≈ 5 ml. The liquid was removed by means of a pipette and the crystals were washed twice with 10 ml of ethyl acetate. The crystals were dried in vacuum to give 1.80 g of pure 1 with a m.p. of $234-235^{\circ}\text{C}$.

Analysis calculated for $\text{C}_6\text{H}_{10}\text{N}_8\text{O}_8$: C, 22.36; H, 3.12; N, 34.78.
Found: C, 22.60; H, 3.20; N, 34.83.

IR (See Appendix A, Figure A-8).

NMR (See Appendix B, Figure B-10).

Mixtures Rich in 3. The crude product was dissolved in 20 ml acetone and filtered to remove insoluble materials. The solution was warmed to reflux and water was added dropwise until the solution remained turbid. More acetone was added and the solution was allowed to cool. The product was collected and washed with acetone. The pure 3 melts at $213-214^{\circ}\text{C}$.

Analysis calculated for $\text{C}_6\text{H}_{10}\text{N}_8\text{O}_8$: C, 22.36; H, 3.13; N, 34.78.
Found: C, 22.67; H, 3.12; N, 35.01.

IR (See Appendix A, Figure A-9).

NMR (See Appendix B, Figure B-11).

Mixture of 1 and 3. In addition, small amounts of mixtures of 1 and 3 can be separated by preparative TLC using 2 mm silica gel plate and a 50:30 THF-hexane solvent system. The R_Fs of 1 and 3 are approximately 0.2 and 0.7, respectively.

MESO-ERYTHRITOL TETRABENZENESULFONATE (23)

Meso-erythritol (12.2 g, 0.1 mole) was added to 200 ml of dry pyridine. This slurry was maintained below 5°C by an ice bath while 75 ml of benzenesulfonyl chloride was added dropwise over a 30-minute period. The cooling bath was removed and the mixture stirred at room temperature for 4 hours. The mixture was then poured onto a mixture of 200 g of ice and 100 ml of concentrated HCl. The product precipitated as an oily solid. This crude product was collected by vacuum filtration and then slurried with 300 ml of methanol. The product was recollected and dried. It weighed 60.2 g (0.088 mole, 88%) and melted at 184-185.5°C.

Analysis calculated for C₂₈H₂₆O₂₄S₄: C, 49.27; H, 3.81.
Found: C, 49.46; H, 3.91.

IR (See Appendix A, Figure A-10).

NMR (See Appendix B, Figure B-12).

ERYTHRO-1,2,3,4-TETRAAZIDOBUTANE (24)

Erythritol tetrabenzenesulfonate (20.4 g, 0.03 mole) and sodium azide were placed in a 500 ml round-bottom flask, dry DMF (200 ml) was added, and the contents were stirred at 110°C for 4 hours. The mixture was cooled and diluted with 300 ml of cold water. The solution was extracted with four 100-ml portions of ether. The ether extracts were combined and both extracted with two 50-ml portions of water. The ether layer was dried over MgSO₄, filtered, and the solvent removed at reduced pressure to give the crude *erythro*-1,2,3,4-tetraazidobutane. No attempt was made to purify the compound because of the known sensitivity of polyazido compounds.

IR (See Appendix A, Figure A-11).

NMR (See Appendix B, Figure B-13).

MESO-1,2,3,4-TETRAAMINO BUTANE (13)

The crude tetraazidobutane was dissolved in 100 ml of 95% ethanol and 1 g of 10% Pd/C was added. This solution was hydrogenated at 55 psi in a Parr apparatus with the tank shut off from the bottle. Every hour the bottle was vented and fresh hydrogen introduced. After 4 hours, the solution was filtered and the solvent removed at reduced pressure to give the crude *erythro*-1,2,3,4-tetraaminobutane. A small portion was reacted with acetic anhydride to give the tetraacetamide with a m.p. of 310-311°C.

Analysis calculated for $C_{12}H_{22}N_4O_4$: C, 50.33; H, 7.75; N, 19.57.
Found: C, 50.29; H, 7.65; N, 19.51.

IR (See Appendix A, Figure A-12).

NMR (See Appendix B, Figure B-14).

TRANS-1,3,5,7-TETRANITROSO-1,3,5,7-TETRAAZADECALIN (11)

The crude *erythro*-1,2,3,4-tetraaminobutane was dissolved in 30 ml of water and this solution was cooled to 5°C. Thirty-seven percent aqueous formaldehyde (4.06 g, 0.06 mole) was then added dropwise with stirring. The cooling bath was removed and the mixture stirred at 50°C for 1 hour. Sodium nitrite (8.28 g, 0.12 mole) was added to the solution. When the sodium nitrite had completely dissolved the solution was cooled to 5°C and a solution of 11.5 g concentrated hydrochloric acid (diluted) 50 ml was added. A precipitate formed almost immediately. After 10 minutes of stirring, the product was collected by vacuum filtration and washed well with water. After drying, the product weighed 2.62 g (0.010 mole, 33%). The material could be recrystallized from DMF/H₂O to give light yellow platelets with a m.p. of 200-202°C (dec).

IR (See Appendix A, Figure A-13).

NMR (See Appendix B, Figure B-15).

TRANS-1,3,5,7-TETRANITRO-1,3,5,7-TETRAAZADECALIN (2)

Fifteen ml of 100% nitric acid was placed in a 50 ml Erlenmeyer flask. A magnetic stirring bar was added and the contents cooled to -30°C by means of a dichloroethane/dry ice slush. *Trans*-1,3,5,7-tetra-nitroso-1,3,5,7-tetraazadecalin was added to this solution over 10 minutes. The dichloroethane/dry ice bath was replaced with an ice water bath. After stirring at 0°C for 30 minutes, the mixture was stirred at 50°C for 10 minutes. The solution was then poured onto 30 g of ice. After the ice had melted the product was collected by vacuum filtration

and was washed well with water. After drying, the crude product weighed 0.66 g. The product was purified by dissolving in warm DMF (60°C) and by adding water until turbid. After cooling to 0°C, the crystals were collected. The purified product weighed 0.41-0.45 g and melted at 252-254°C.

Analysis calculated for $C_6H_{10}N_8O_8$: C, 22.36; H, 3.13; N, 34.78.
Found: C, 22.62; H, 3.21; N, 34.62.

IR (See Appendix A, Figure A-14).

NMR (See Appendix B, Figure B-16).

MESO-4,4'-(1,3-DINITRO-1,3-DIAZACYCLOPENTANE) (4)

By diluting the mother liquors from the recrystallization of 2 with water, an impure material could be isolated. This material could be purified by preparative TLC (silica gel G_1 /THF-hexane) to give pure 4 with a m.p. of 198-199°C.

Analysis calculated for $C_6H_{10}N_8O_8$: C, 27.36; H, 3.13; N, 34.78.
Found: C, 27.52; H, 3.22; N, 34.82.

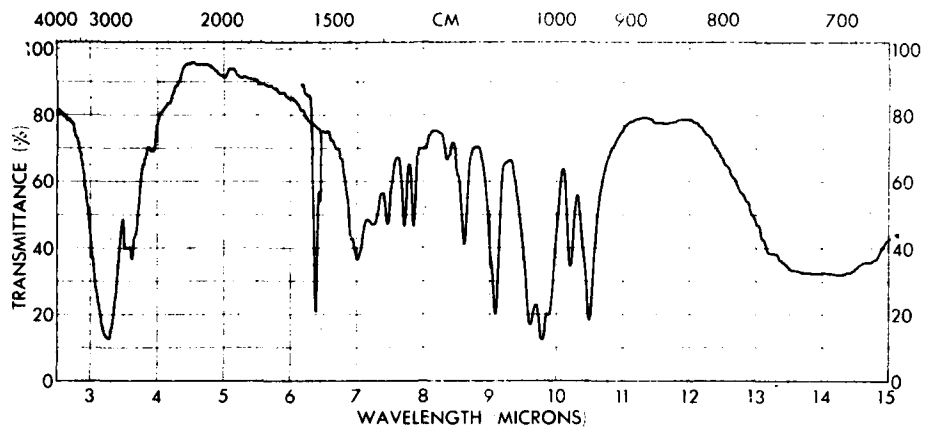
IR (See Appendix A, Figure A-15).

NMR (See Appendix B, Figure B-17).

NWC TP 6416

Appendix A
INFRARED SPECTRA OF COMPOUNDS

NWC TP 6416

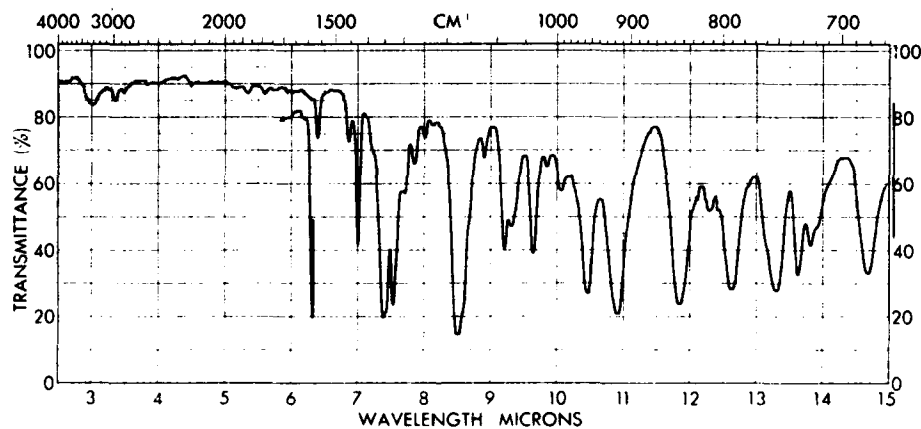


SPECTRUM NO. 1001-38-1	ORIGIN	LEGEND <i>poly</i>	REMARKS
SAMPLE		1.	
<chem>OC[C@H](O)[C@@H](O)[C@@H](O)CO</chem>	PURITY	2.	
L-Threitol	PHASE <i>KB</i>	DATE	
m.p. 86-87°C	THICKNESS	OPERATOR <i>W. Allen</i>	

PART NO. 137-1281

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-3. IR Spectrum of L-Threitol (14).

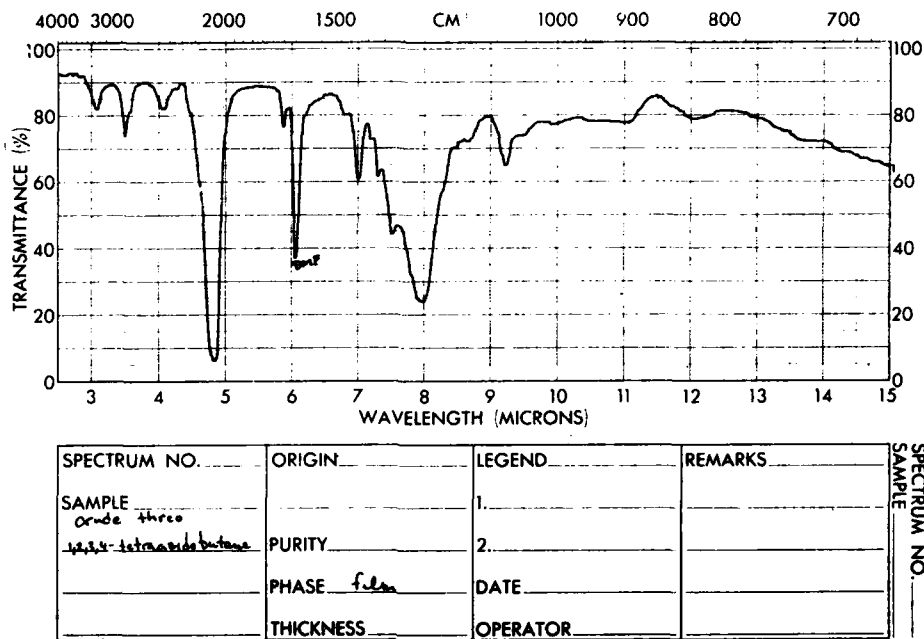


SPECTRUM NO. 1001-40-1	ORIGIN	LEGEND	REMARKS
SAMPLE		1.	
L-Threitol tetrabenzene sulfonate	PURITY	2.	
<i>Benzenesulfonate</i>	PHASE	DATE <i>June 10, 1962</i>	
	THICKNESS	OPERATOR <i>W. Allen</i>	

PART NO. 137-1281

THE PERKIN-ELMER CORPORATION NORWALK, CONN.

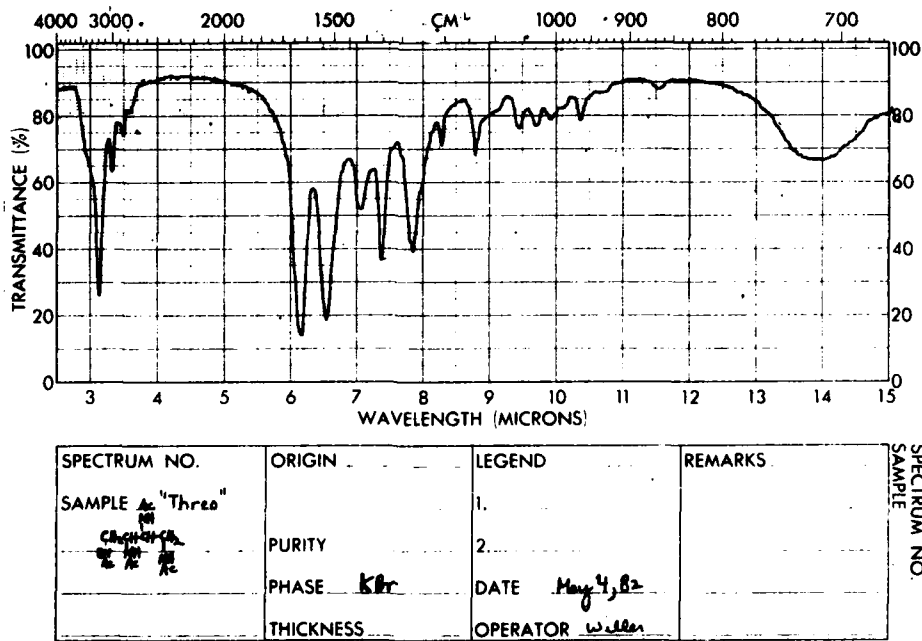
FIGURE A-4. IR Spectrum of L-Threitol-tetrabenzenesulfonate (18).



PART NO. 137-1281-1A *

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-5. IR Spectrum of *Threo*-1,2,3,4-tetraazidobutane (19).

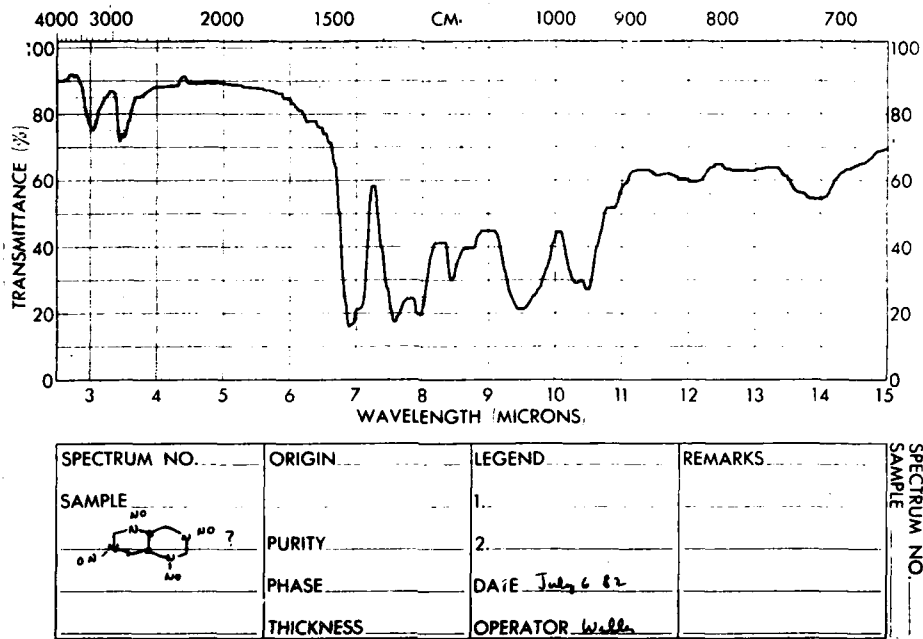


PART NO. 137-1281-1A *

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-6. IR Spectrum of *Threo*-1,2,3,4-tetraacetamidobutane (10b).

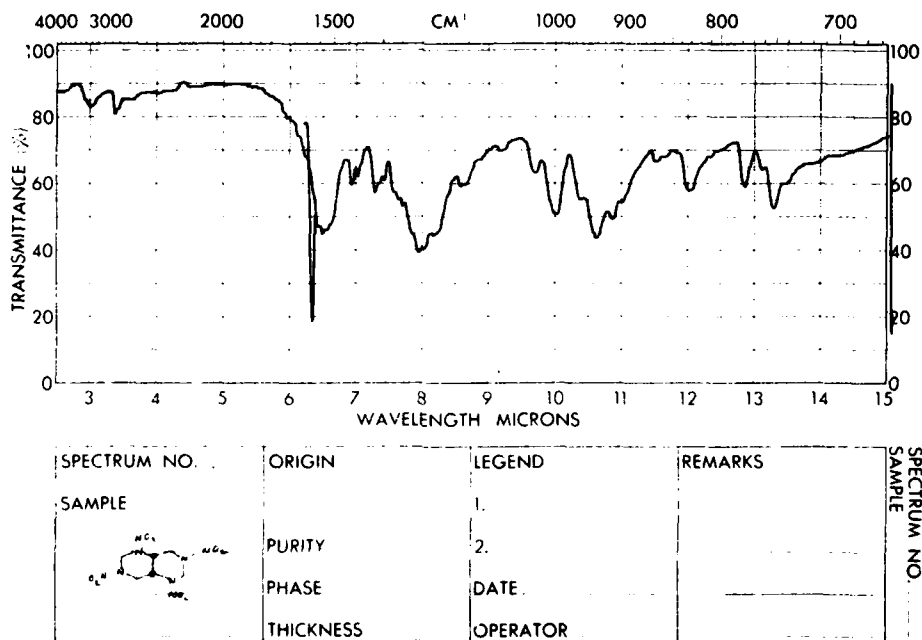
NWC TP 6416



PART NO. 137-1281 IN *

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-7. IR Spectrum of a Mixture of *Cis*-1,3,5,7-tetranitroso-1,3,5,7-tetraazadecalin (8) and *D*-(4,4')-bi-(1,3-dinitroso-1,3-diazacyclopentane) (22).

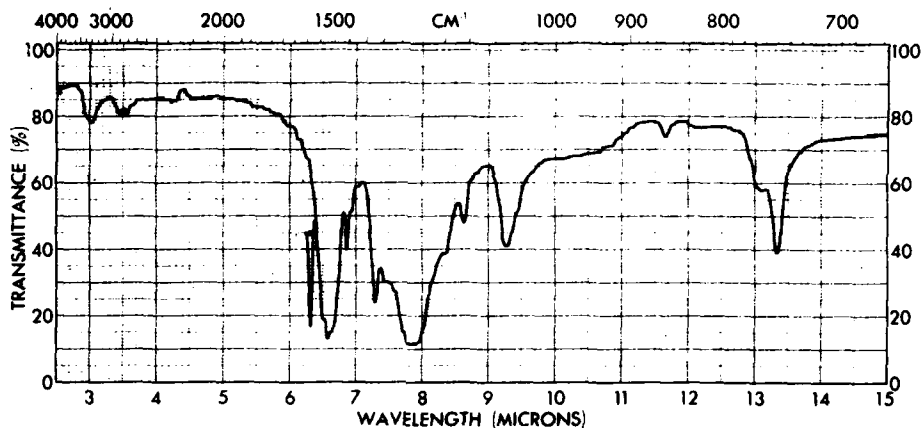


PART NO. 137 24

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FIGURE A-8. IR Spectrum of *Cis*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin (1).

NWC TP 6416

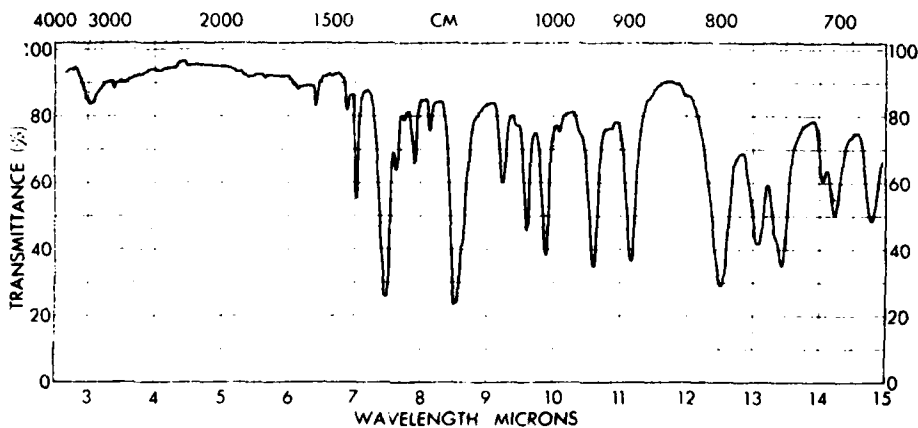


SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE		1.	
	PURITY	2.	
	PHASE	DATE	
	THICKNESS	OPERATOR	

PART NO. 137-1281 PL. ©

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FIGURE A-9. IR Spectrum of *D*-4,4'-(1,3-dinitro-1,3-diazacyclopentane) (3).



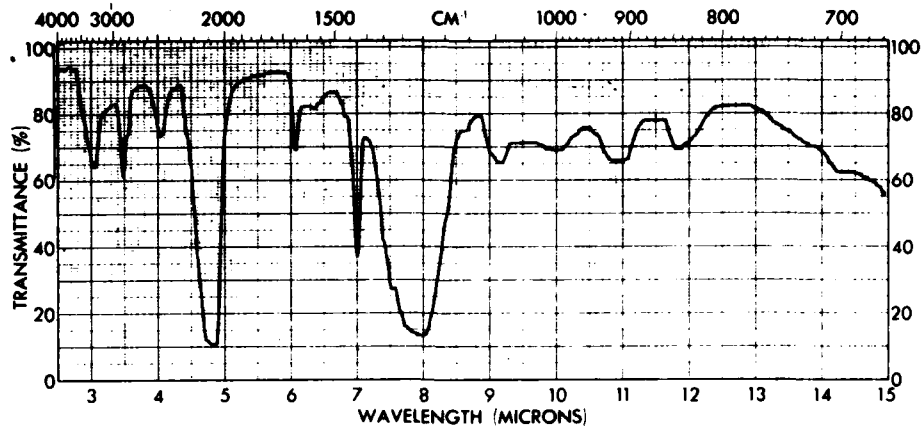
SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE		1.	
	PURITY	2.	
	PHASE KBr	DATE July 19, 52	
	THICKNESS	OPERATOR Miller	

PART NO. 137-1281

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-10. IR Spectrum of *Meso*-erythritol Tetrabenzene-sulfonate (23).

NWC TP 6416



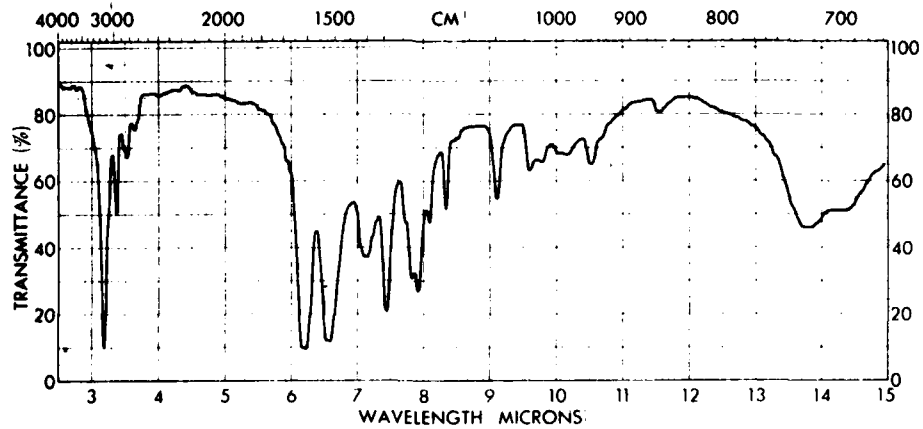
SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE <i>Erythro-</i>		1.	
<i>1,2,3,4-tetraazido-</i>	PURITY	2.	
<i>butane</i>	PHASE <i>liquid</i>	DATE <i>July 20, 52</i>	
	THICKNESS	OPERATOR <i>Miller</i>	

SPECTRUM NO. SAMPLE

PART NO 137-1281

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-11. IR Spectrum of *Erythro*-1,2,3,4-tetraazadibutane (24).



SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE		1.	
<i>Meso-</i>	PURITY	2.	
<i>1,2,3,4-tetraacetamido-</i>	PHASE <i>liquid</i>	DATE <i>July 20, 52</i>	
<i>butane</i>	THICKNESS	OPERATOR <i>Miller</i>	

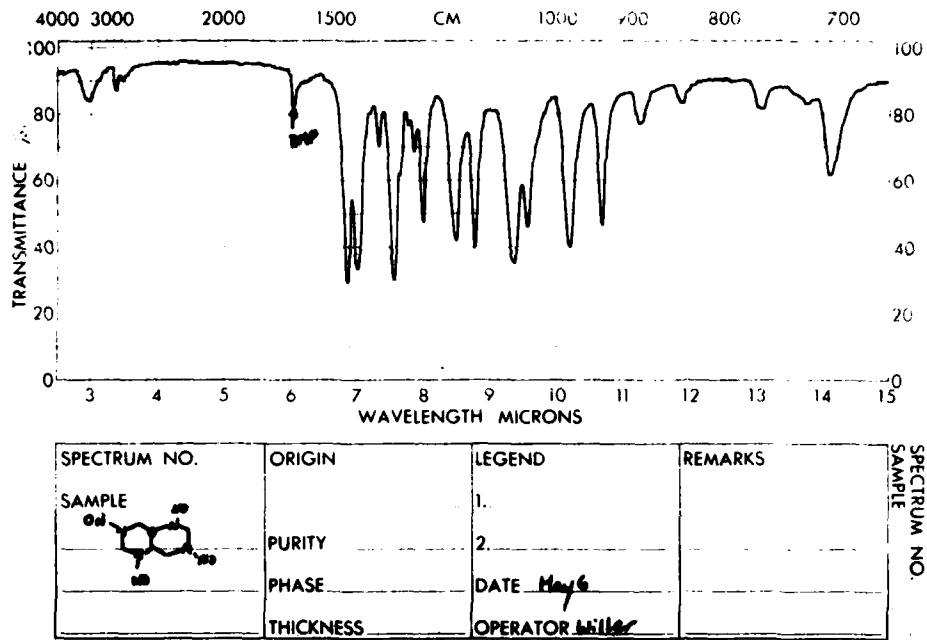
SPECTRUM NO. SAMPLE

PART NO 137-1281

THE PERKIN-ELMER CORPORATION, NORWALK, CONN.

FIGURE A-12. IR Spectrum of *Meso*-1,2,3,4-tetraacetamidobutane (13b).

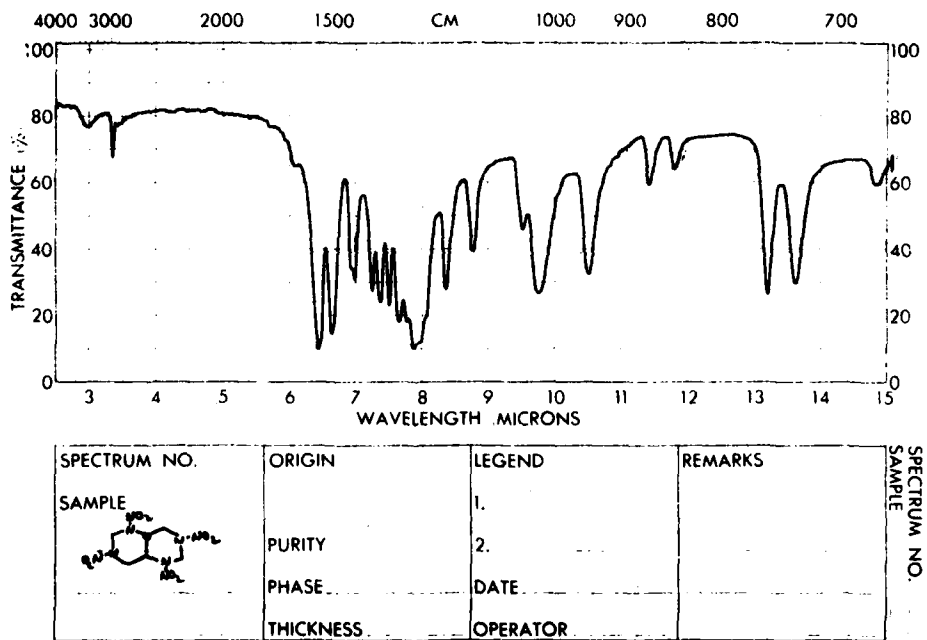
NWC TP 6416



PART NO 137-1281

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FIGURE A-13. IR Spectrum of *Trans*-1,3,5,7-tetranitroso-1,3,5,7-tetraazadecalin (1).



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FIGURE A-14. IR Spectrum of *Trans*-1,3,5,7-tetranitro-1,3,5,7-tetraazadecalin (2).

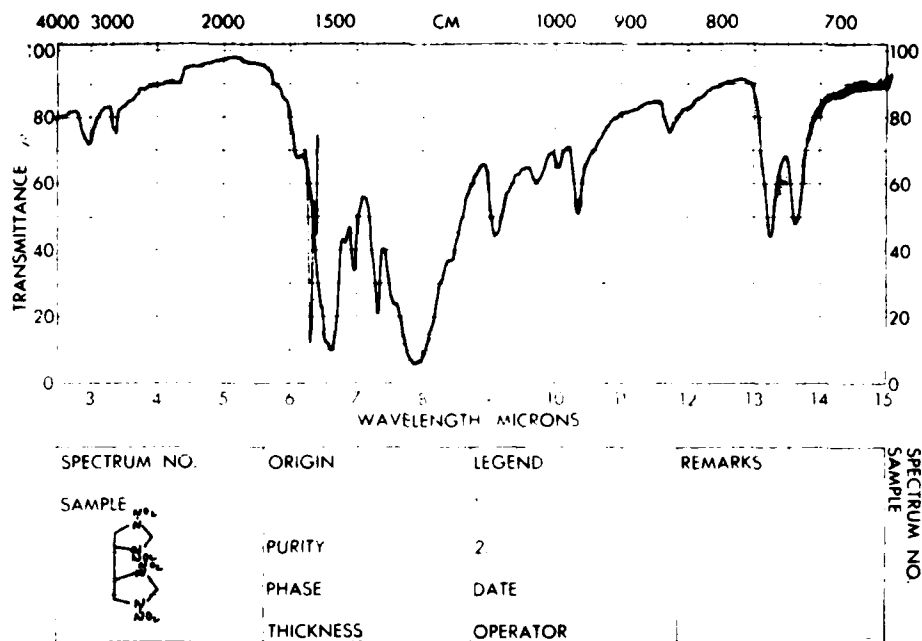


FIGURE A-15. IR Spectrum of *Meso*-4,4'-(1,3-dinitro-1,3-diazacyclopentane) (4).

NWC TP 6416

Appendix B

^1H NMR SPECTRA OF COMPOUNDS

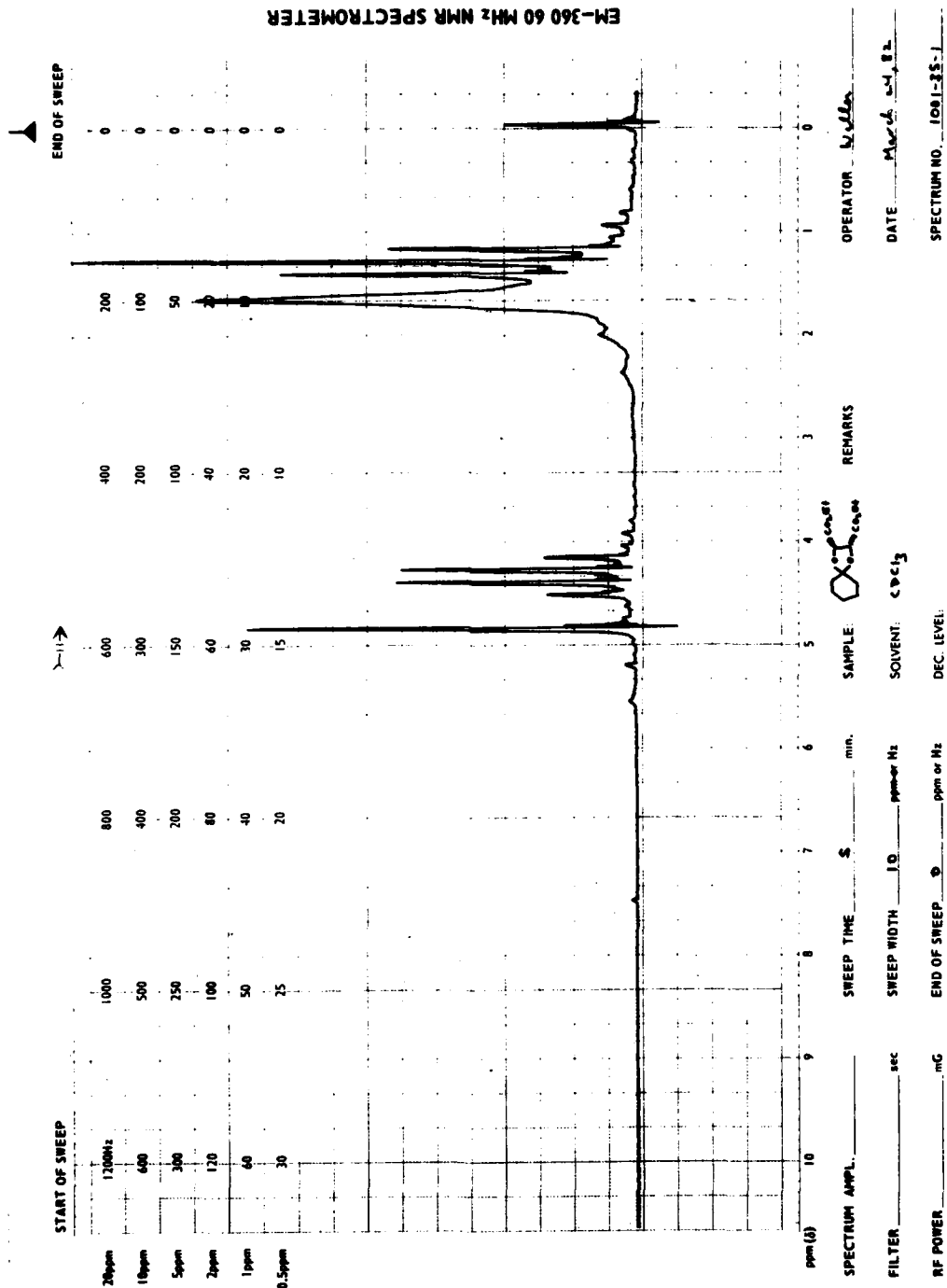


FIGURE B-1. NMR Spectrum of Diethyl-2,3-O-cyclohexylidene-L-tartrate (16).

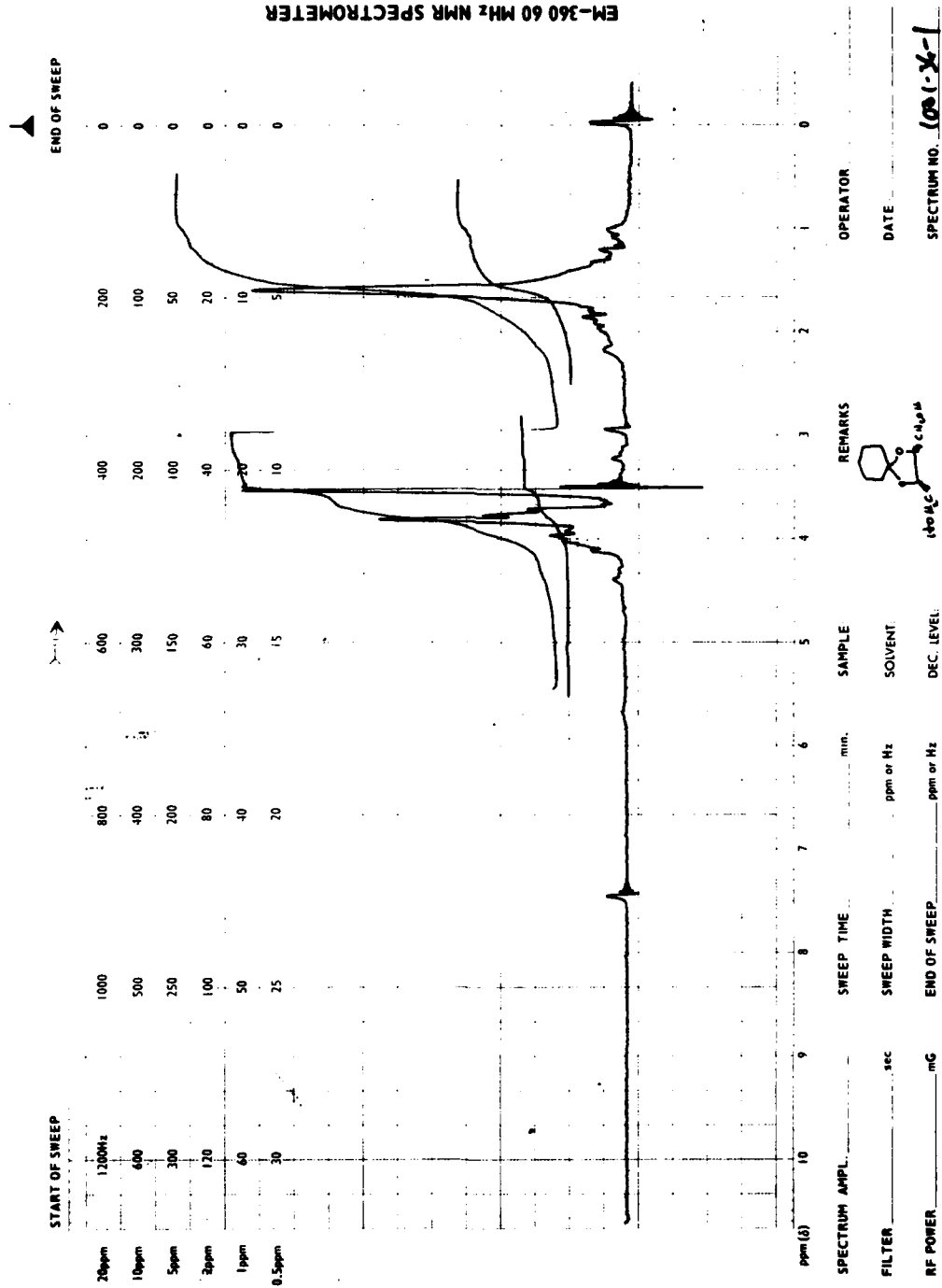


FIGURE B-2. NMR Spectrum of 2,3-O-Cyclohexylidene-L-threitol (17).

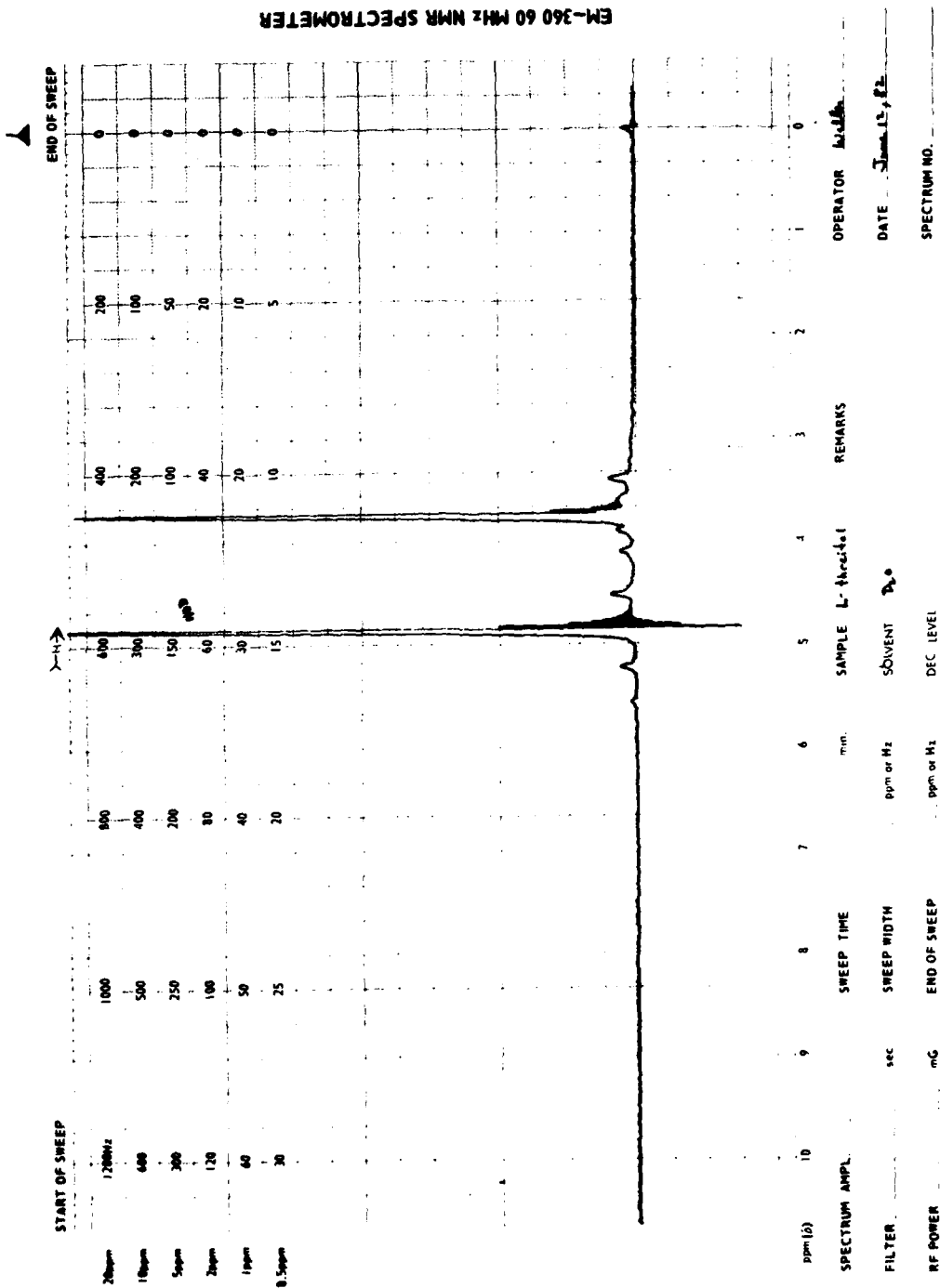


FIGURE B-3. NMR Spectrum of L-Threitol (14).

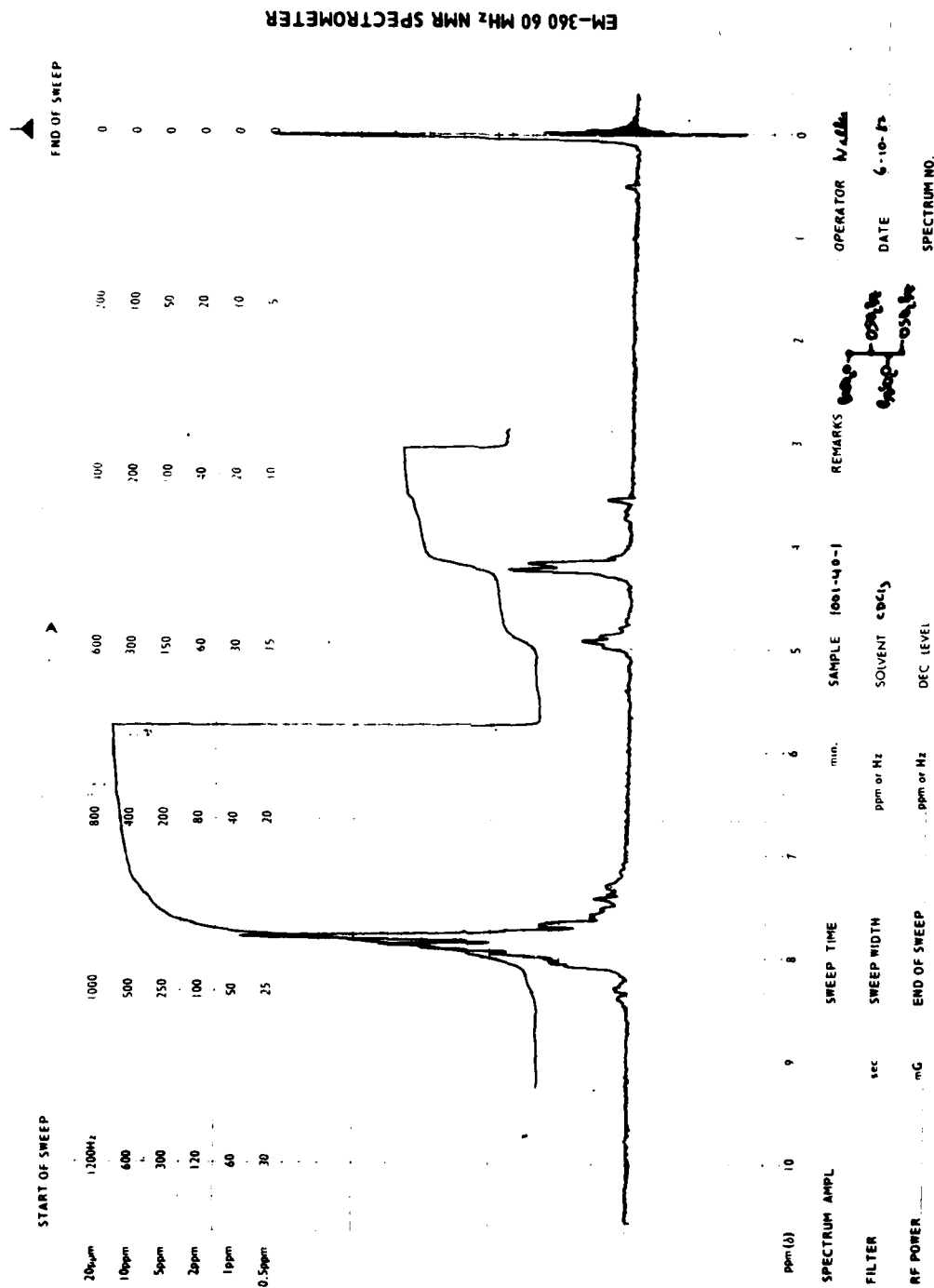


FIGURE B-4. NMR Spectrum of L-Threitol-tetrabenzenesulfonate (18).

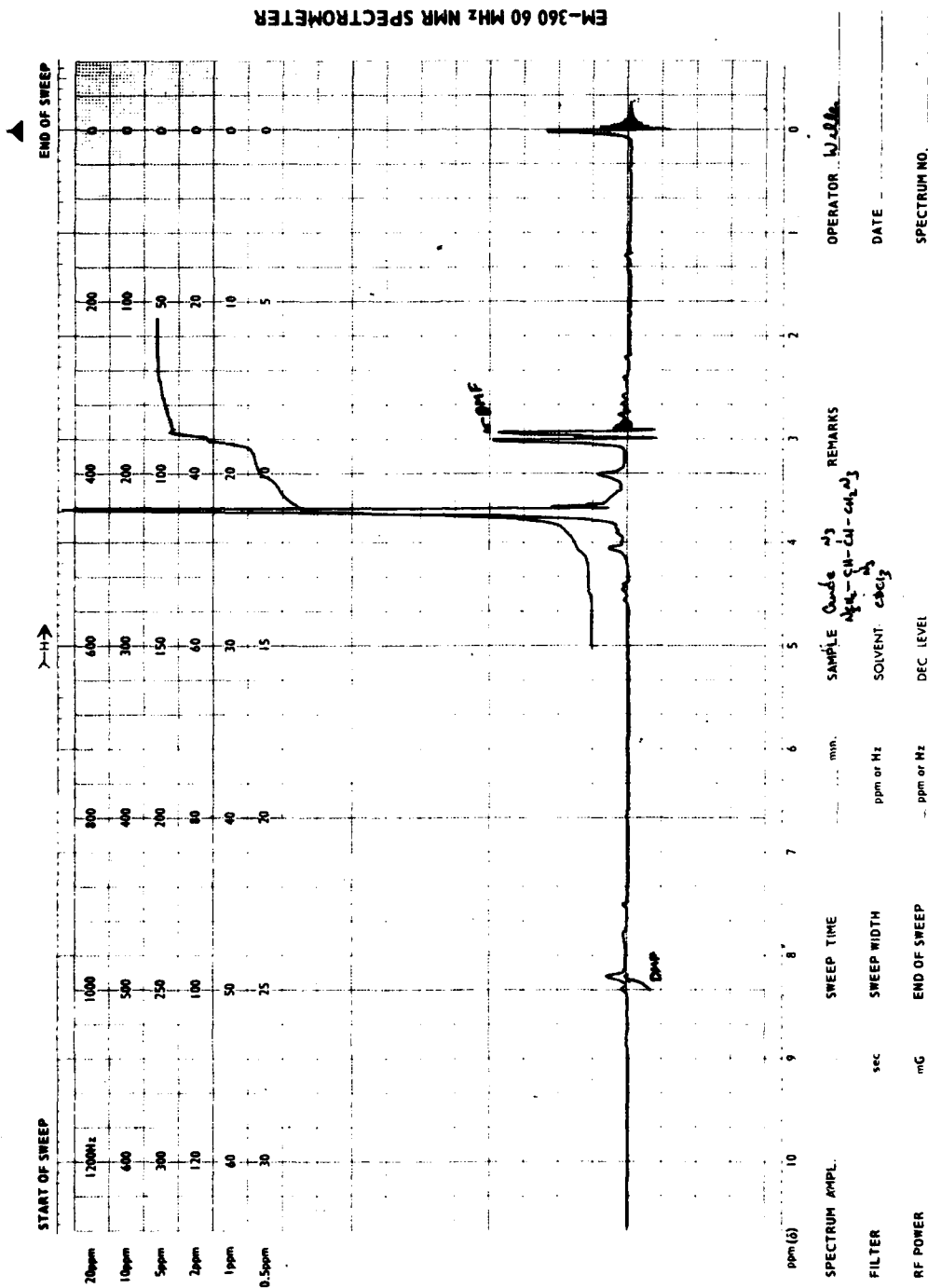


FIGURE B-5. NMR Spectrum of Threo-1,2,3,4-tetraazidobutane (19).

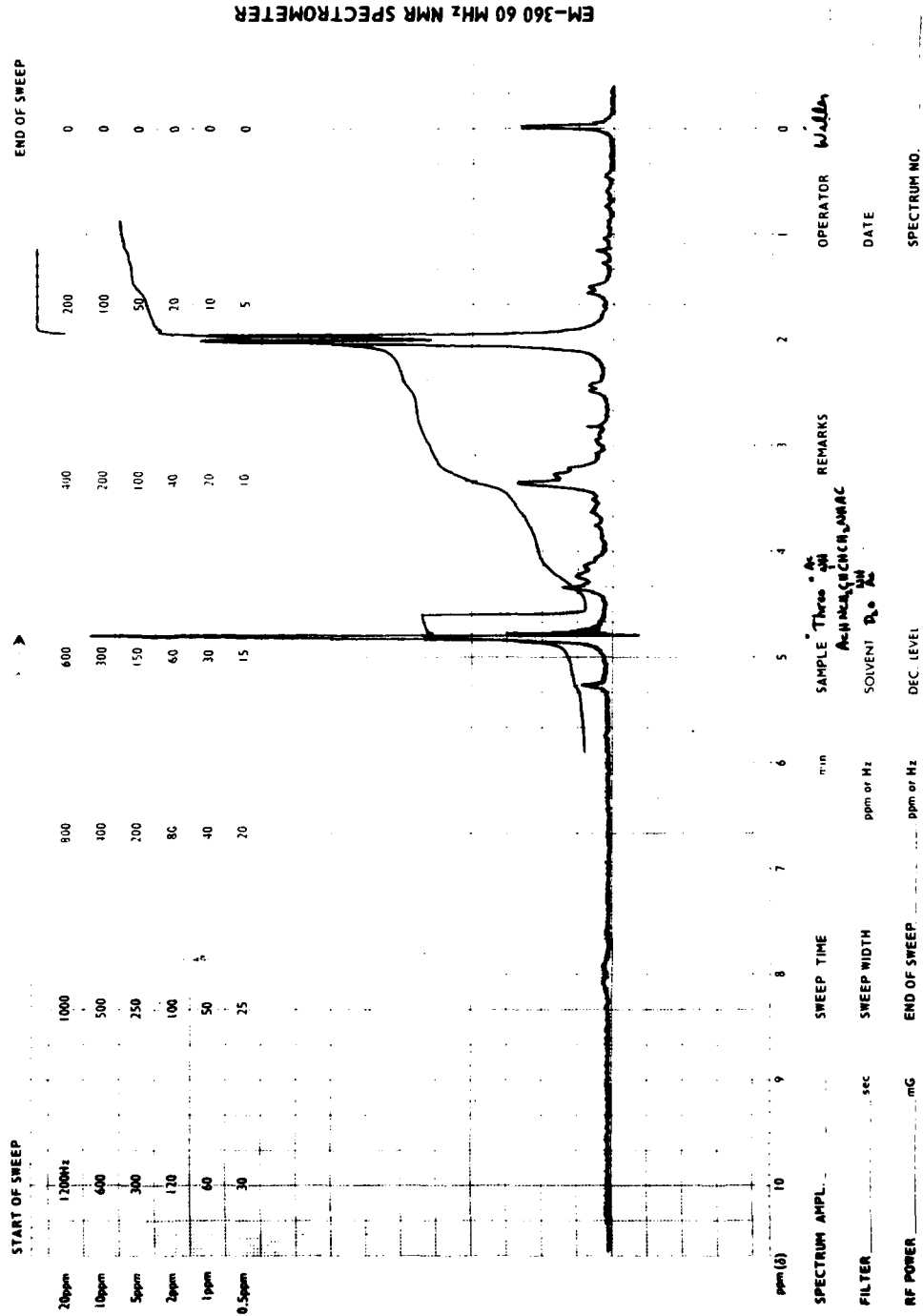


FIGURE B-6. NMR Spectrum of Threo-1,2,3,4-tetraacetamidobutane (10b).

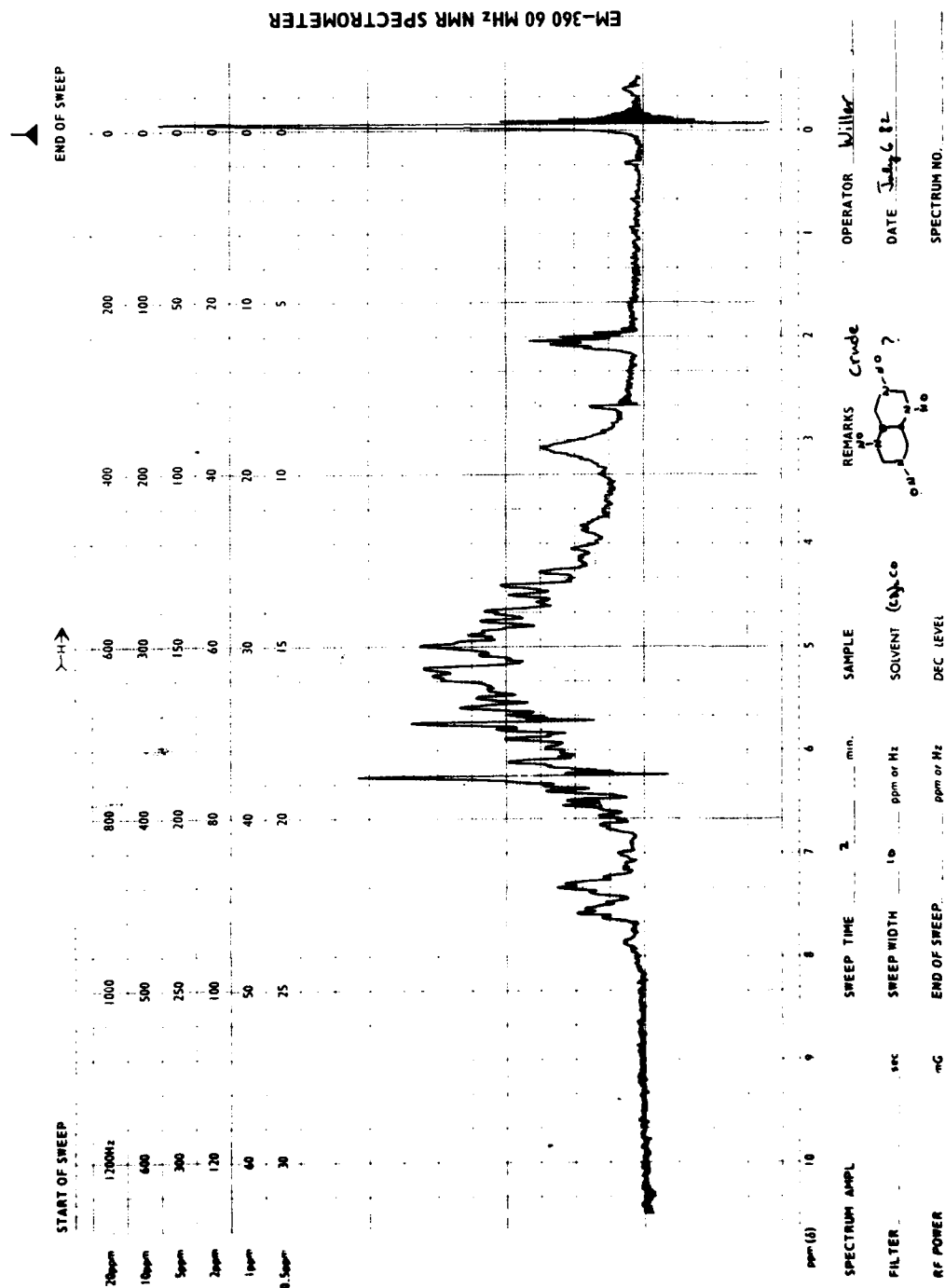


FIGURE B-7. NMR Spectrum of a Mixture of (1S-1,3,5,7-tetranitroso-1,3,5,7-tetra-azadecalin (8) and D-(4,4')-bi-(1,3-dinitroso-1,3-diazacyclopentane) (22)).

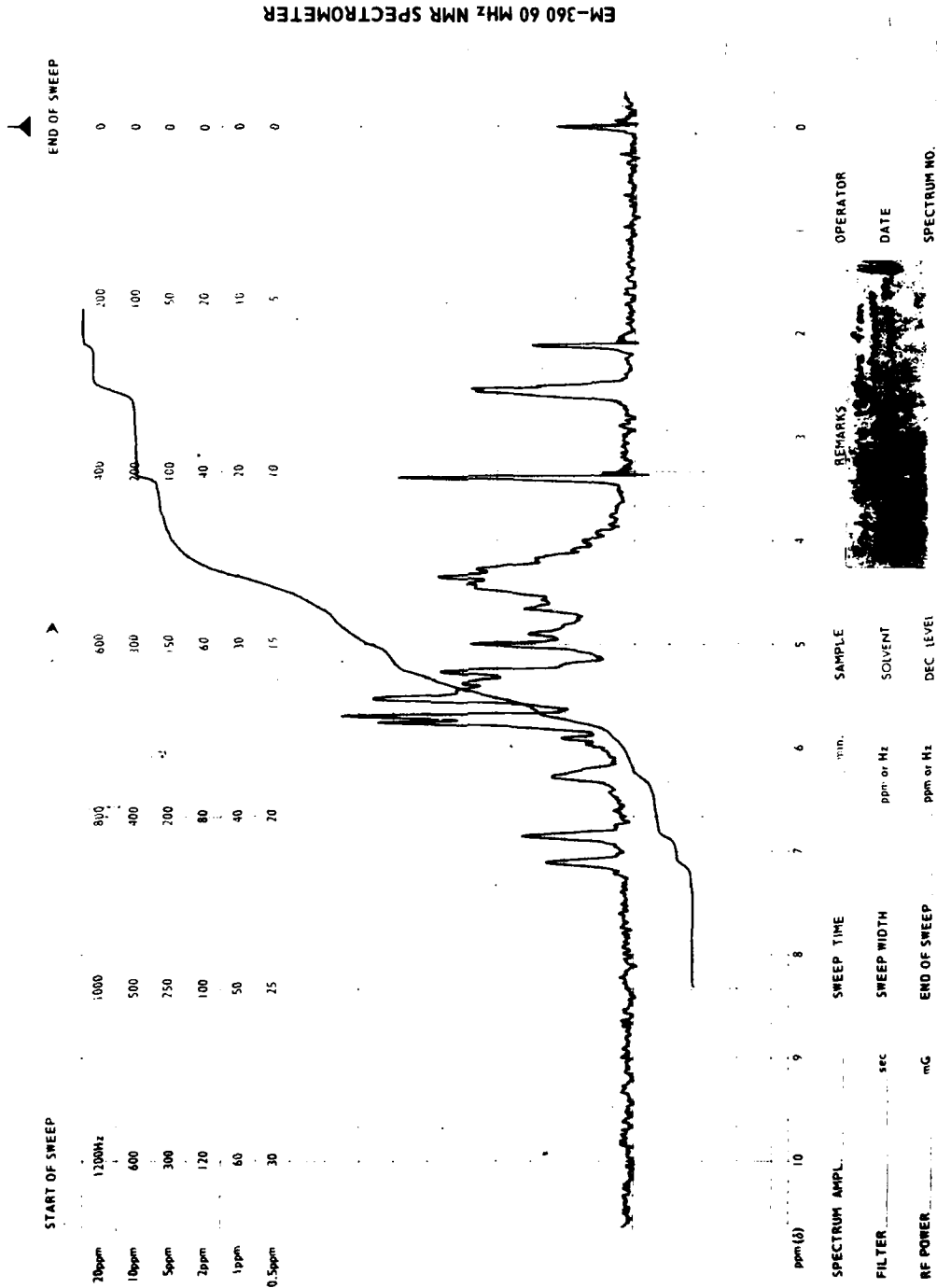


FIGURE B-8. NMR Spectrum of the Crude Product From the Nitrolysis of a Mixture of 2,6-1,3,5,7-tetranitroso-1,3,5,7-tetraazadecalin (8) and D-(4,4')-bi-(1,3-dinitroso-1,3-diazacyclopentane) (22) which is Rich in 7-4,4'-1,3-dinitro-1,3-diazacyclopentane (3).

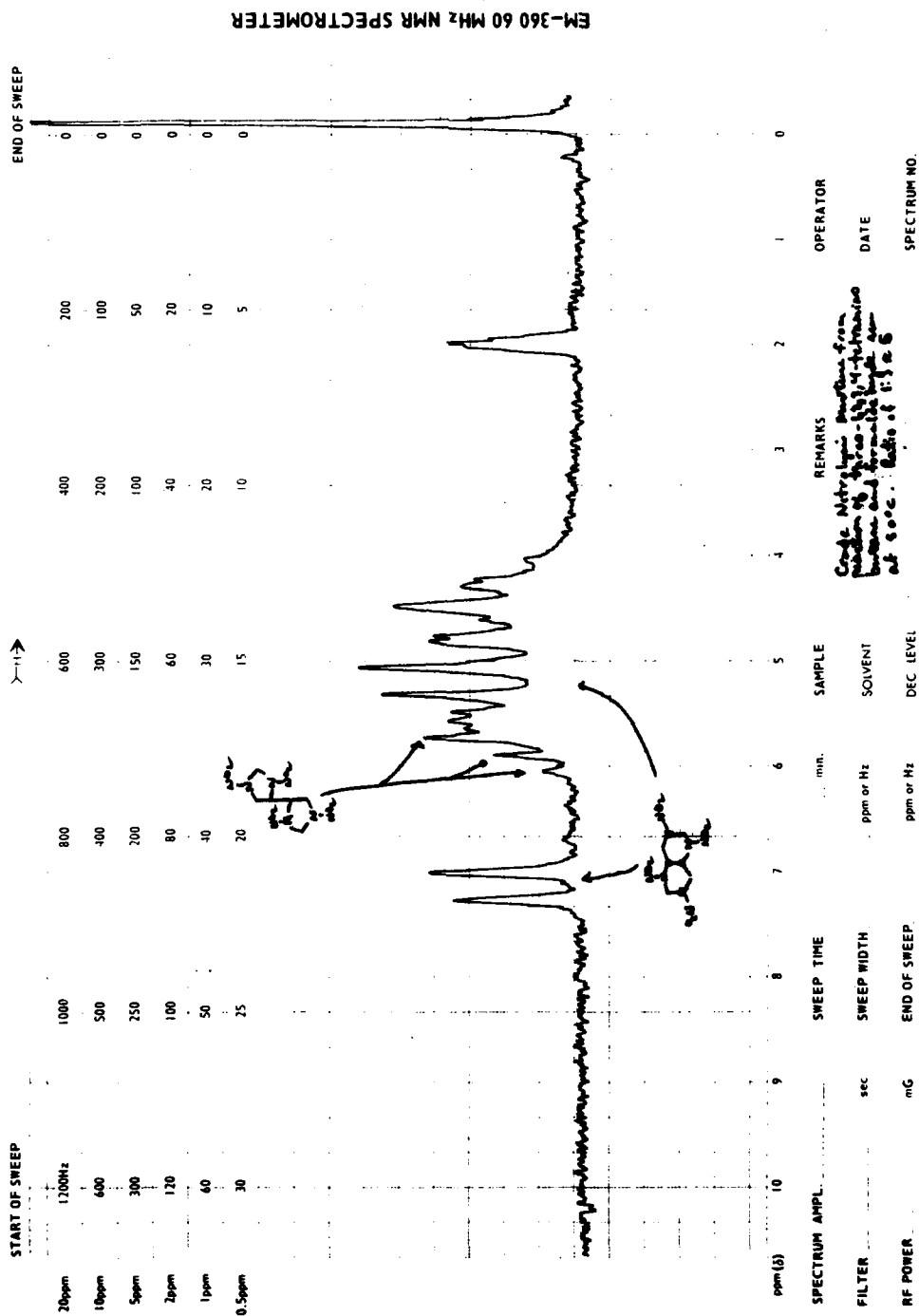


FIGURE B-9. NMR Spectrum of the Crude Product From the Nitrolysis of a Mixture of *Cis*-1,3,5,7-tetraazadecalin (8) and *D*-(4,4')-bi-(1,3-dinitroso-1,3-diazacyclopentane) (22) which is Rich in *Cis*-1,3,5,7-tetraazadecalin (8).

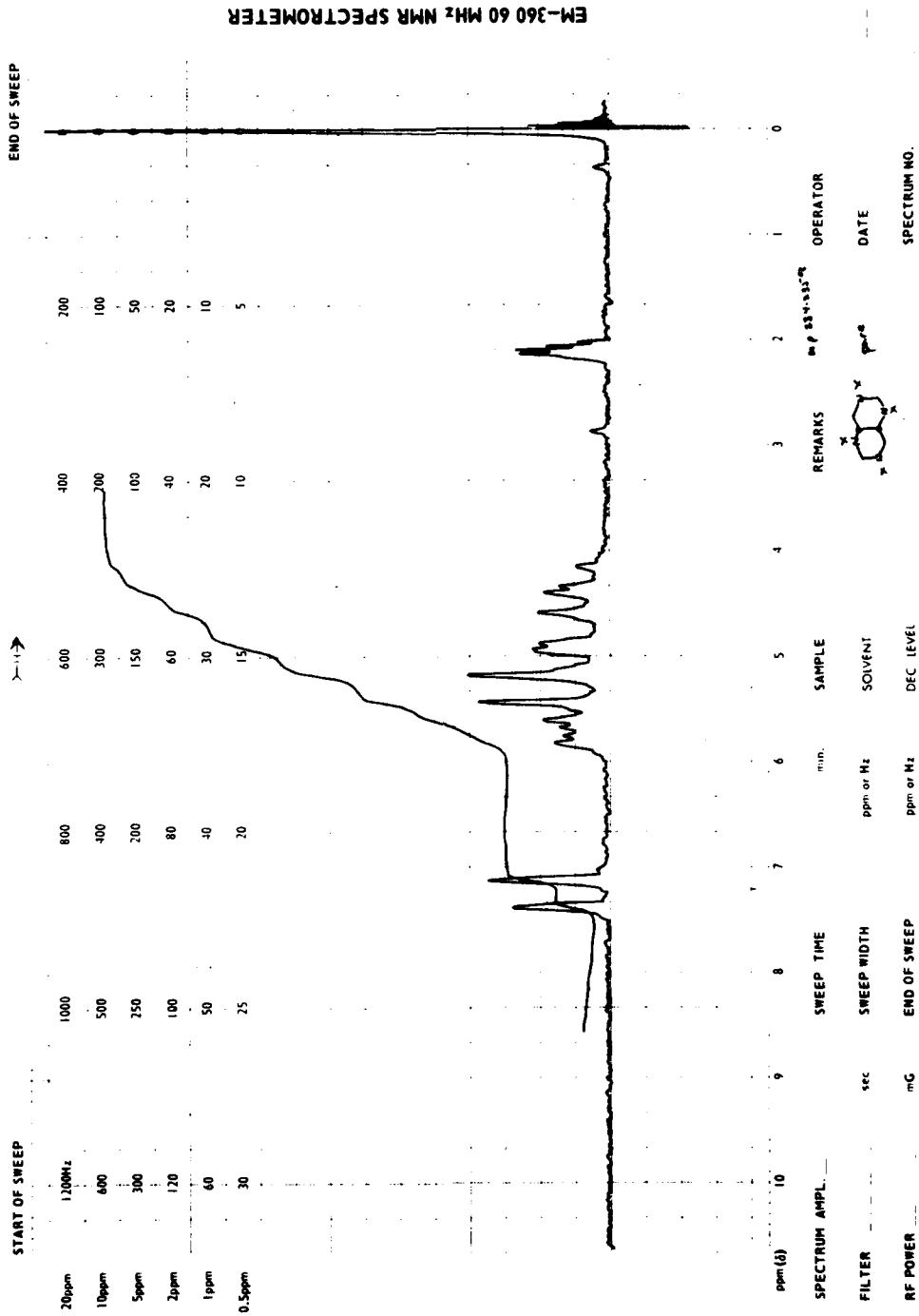


FIGURE B-10. NMR Spectrum of *Cir*-1,3,5,7-tetraazadecalin (I).

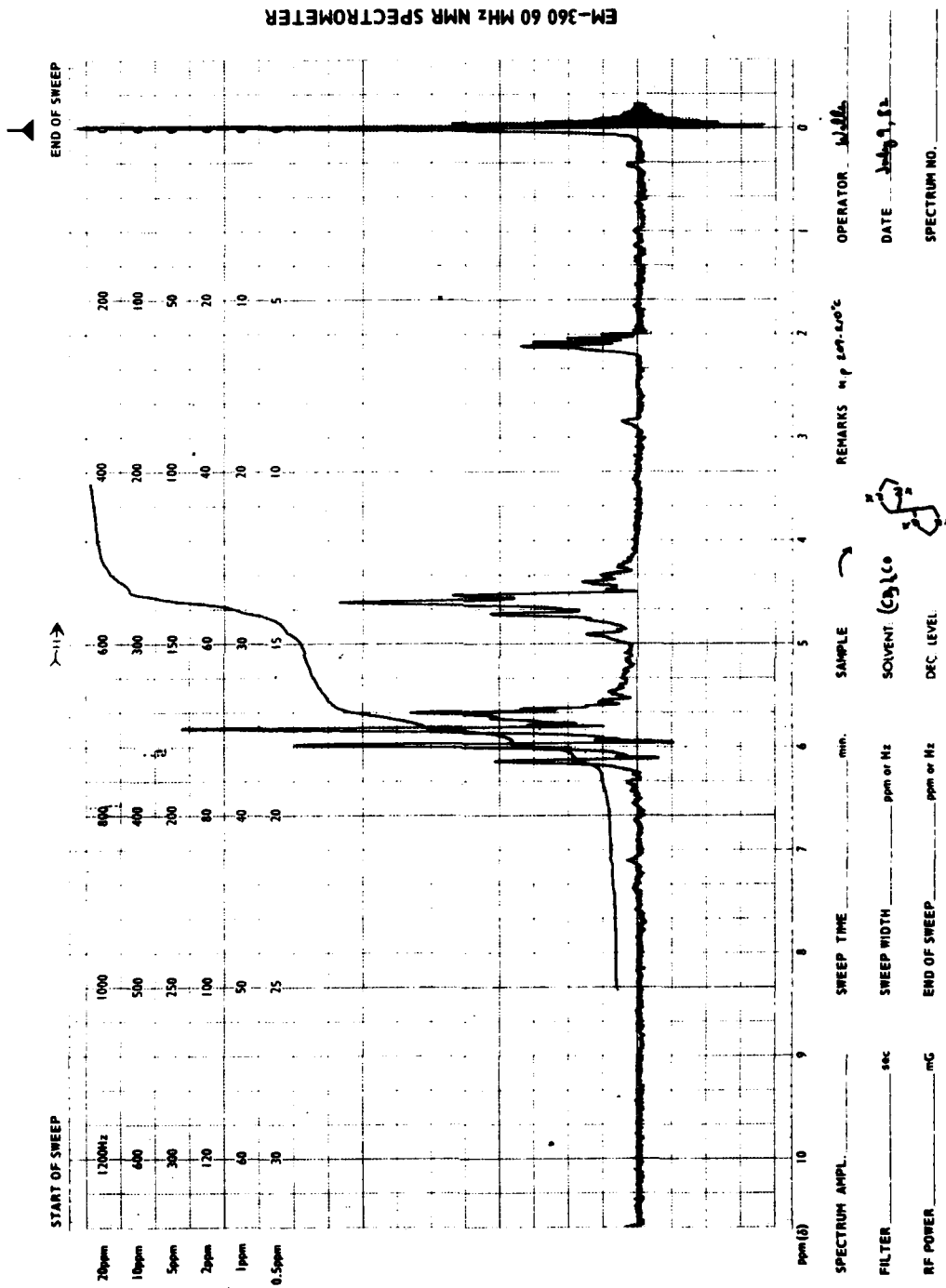


FIGURE B-11. NMR Spectrum of D-4,4'-(1,3-dinitro-1,3-diazacyclopentane) (3).

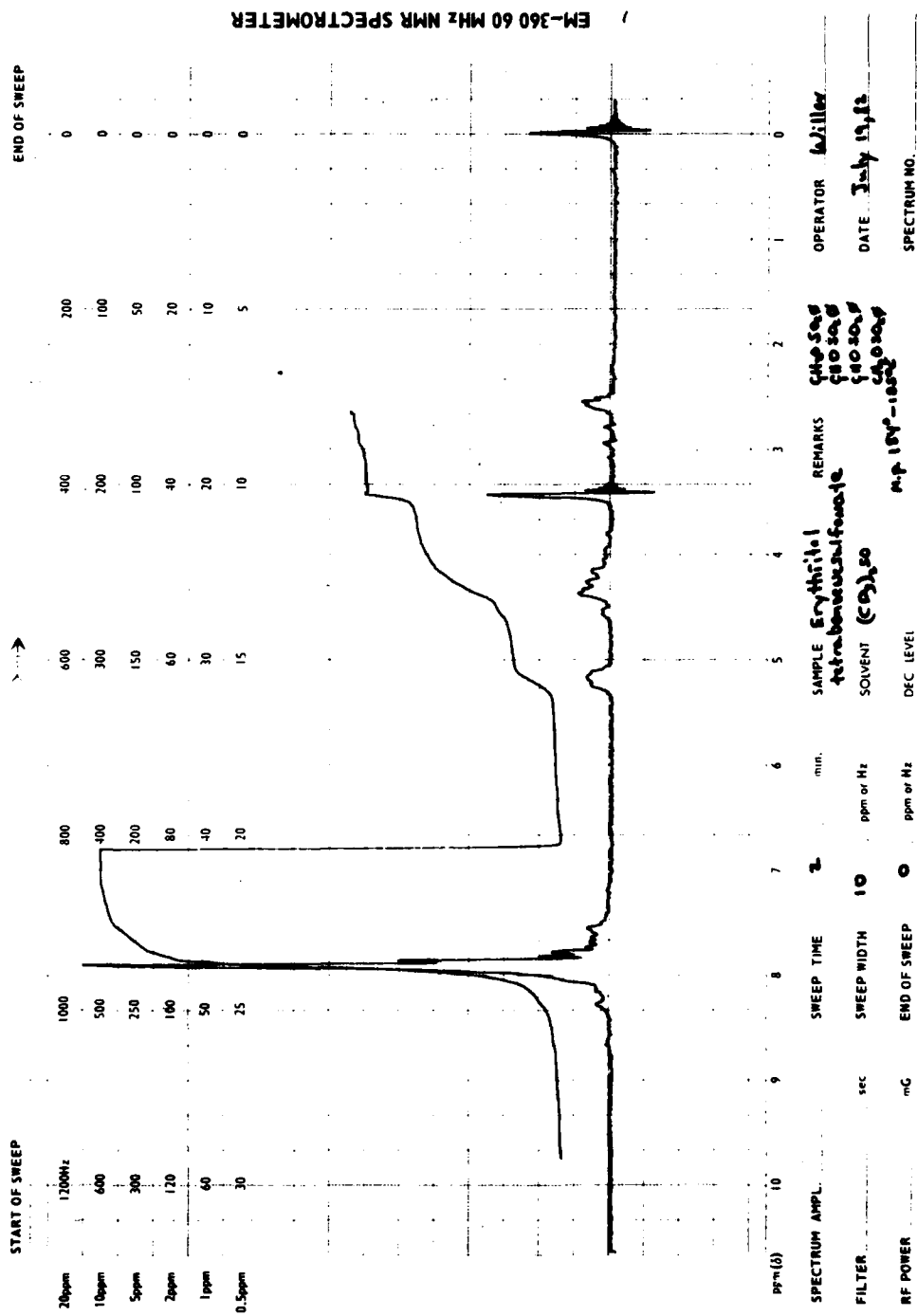


FIGURE B-12. NMR Spectrum of *Meso*-erythritol Tetrabenzenesulfonate (23).

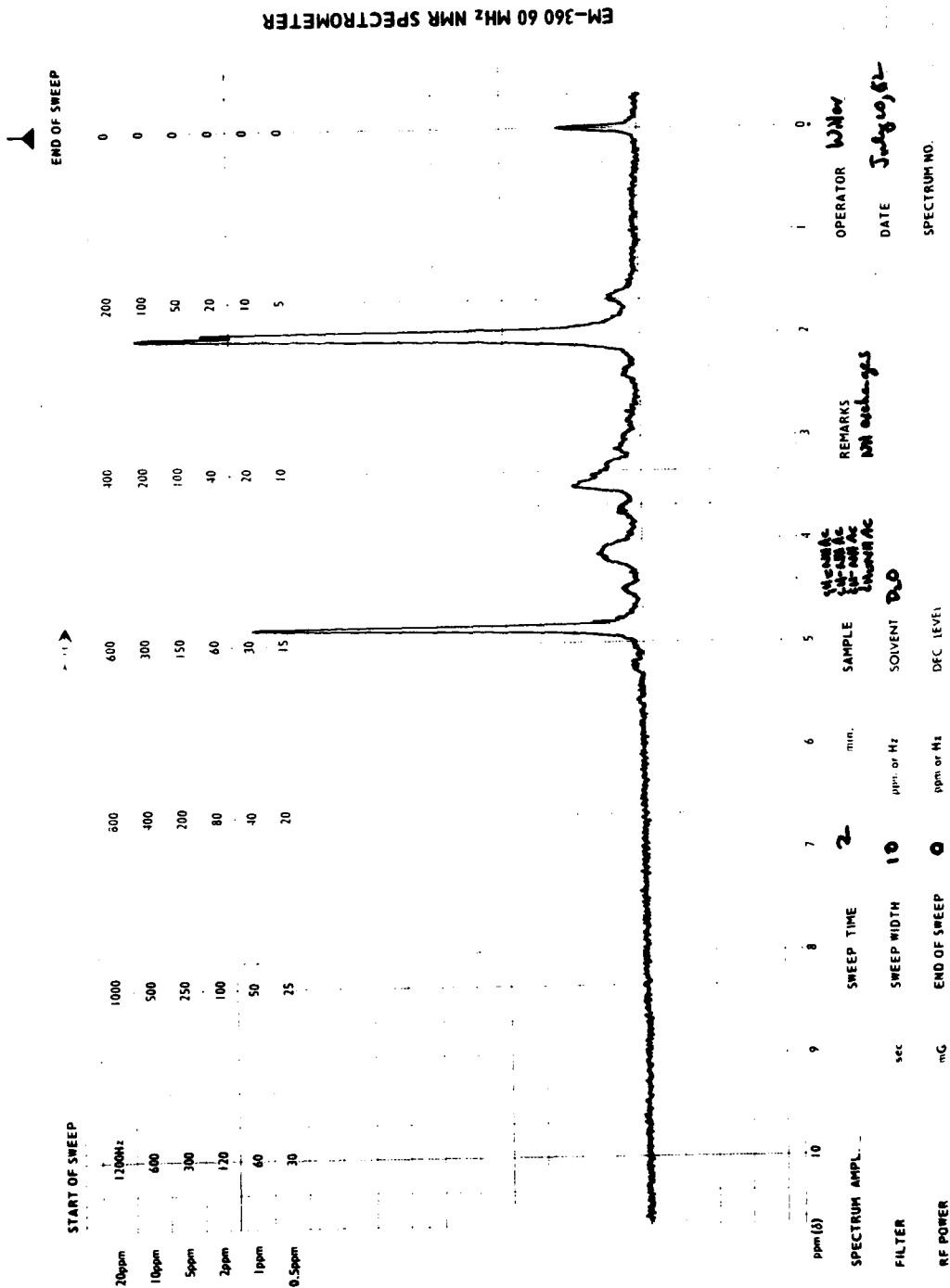


FIGURE B-14. NMR Spectrum of MesO-1,2,3,4-tetraacetamidobutane (13b).

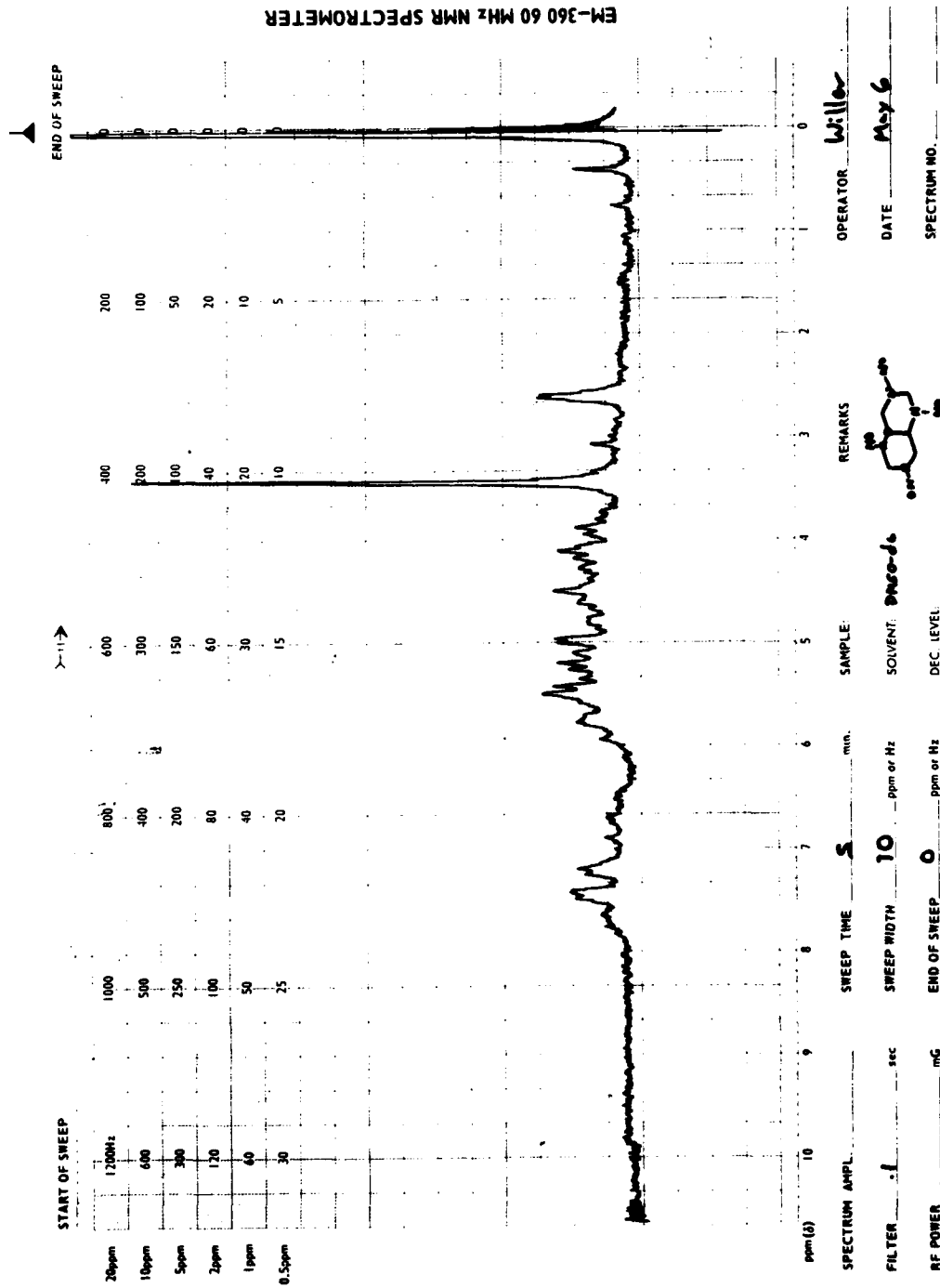


FIGURE B-15. NMR Spectrum of *Trans*-1,3,5,7-tetraazadecalin (II).

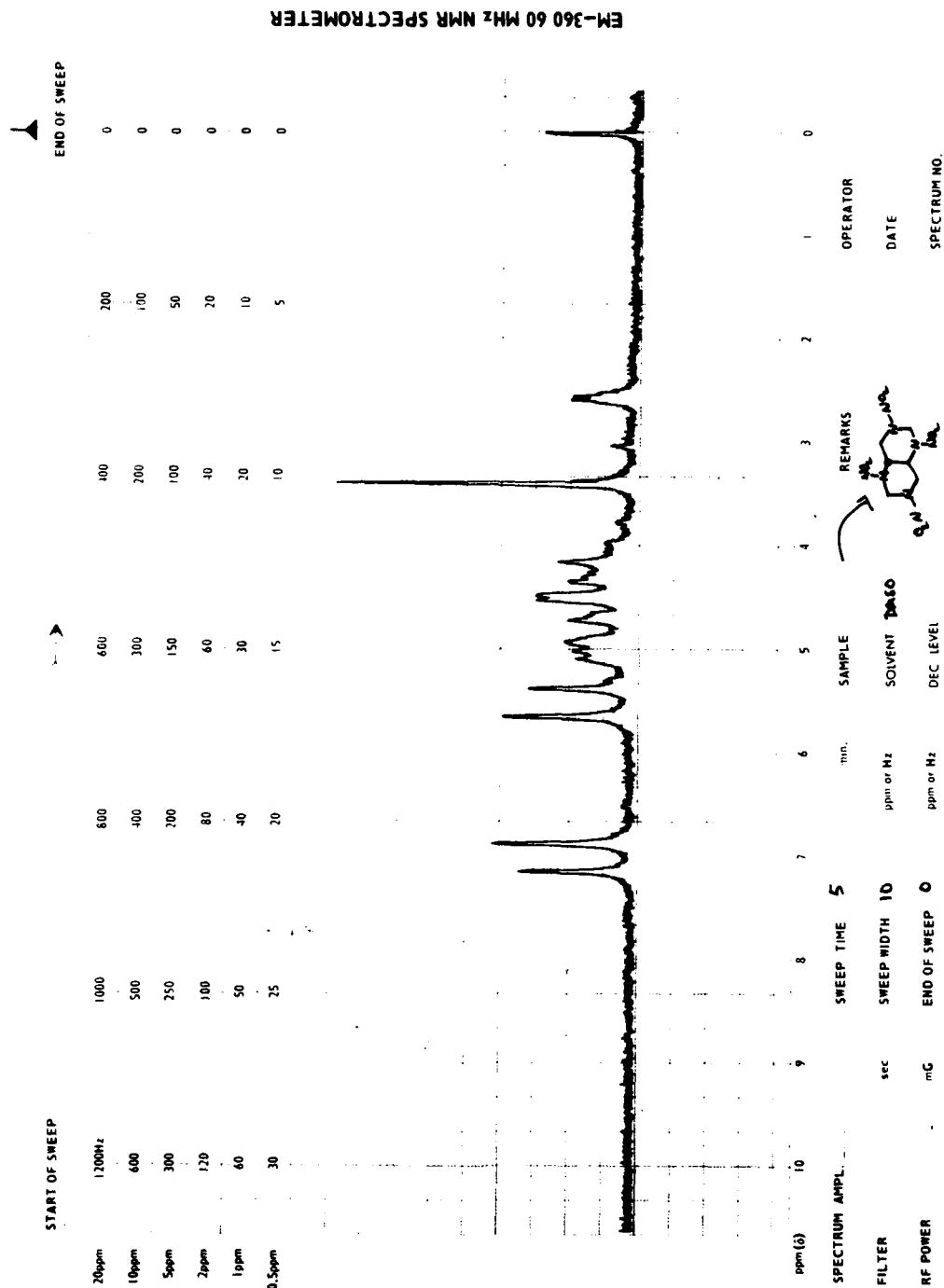


FIGURE B-16. NMR Spectrum of *Trans*-1,3,5,7-tetraazadecalin (2).

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